Nature and Distribution of Sulfur in Five Soil Profiles Correlated with Plant Responses

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A DISSERTATION PRESENTED TO THE GRADUATE COUNCIL OF
THE UNIVERSITY OF FLORIDA
IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE
DEGREE OF DOCTOR OF PHILOSOPHY

UNIVERSITY OF FLORIDA
August, 1955

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ACKNOWLEDGMENTS

Deepest appreciation is given to Dr. F. B. Smith, Head of the Soils Department, whose administrative efficiency and personal attention has made this achievement possible for me.

To Dr. J. R. Neller, Chairman of the Supervisory Committee, I am fully indebted especially for his eminent guidance, supervision, and encouragement throughout the duration of this work.

Sincere appreciation goes to Dr. George D. Thornton who ably assisted in planning and supervising the course work and to Doctors L. C. Hammond, T. W. Stearns, David Duncan, D. E. McCloud, and W. D. Hanson for serving as Supervisory Committee members.

I am sincerely grateful for the contribution from Dr. C. F. Eno who so willingly gave his time and personal equipment in taking the majority of the photographs.

Recognition is also made for the consultation and encouragement from the staff members of the Soils Department in the Agricultural Experiment Station.

My kindest appreciation is felt for my wife, Dorothy, for encouragement, patience, devotion, and consideration, and to my sons who made this work and achievement more meaningful.

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INTRODUCTION

The soil has been found to be deficient in sulfur in many areas of the United States and in foreign countries for normal plant growth. Too little attention has been directed toward sulfur as a plant nutrient and as a fertilizer material particularly in some regions where it is likely to be deficient in the soil.

Nitrogen, phosphorus, and potassium have received practically all the emphasis in fertilizer practices. Recent publications indicate a large increase in the amount of soil research and attention given to copper, zinc, molybdenum, boron, manganese, magnesium, cobalt, iron, aluminum, calcium, and sodium. When the amount and importance of sulfur in plants and its mobility in soils are considered, it is readily realized that sulfur in the soil as a plant nutrient should be investigated more thoroughly. This need is amplified by the use of increasing amounts of high analysis fertilizers which contain only small quantities of sulfur.

This experimental work was conducted in the Soils Department of the University of Florida's Agricultural Experiment Station. The objectives of this work were: (1) to determine whether or not native sulfur in Florida soils is

sufficient for normal growth of plants; if not, to determine the rate at which sulfur must be added to produce normal growth of plants on these soils; (2) to critically study and evaluate the methods used in measuring the amounts of extractable sulfur in soils; (3) to determine the nature and distribution of sulfur in the profiles of certain soils and to correlate the plant responses with chemical analyses of the soils; and (4) to make calculations for sulfur exhaustion studies on these soils.

REVIEW OF LITERATURE

It is impossible to trace the first use of sulfur as a fertilizer, either as gypsum or as other forms. Sulfur was added in manures and plant residues long before chemists knew that this element was present in these materials. Probably the first use of sulfur in North American agriculture was carried out by the Indians when they buried fish in the soil before they planted corn since fish contain large amounts of sulfur.

Alway (3) divides the history of sulfur as a fertilizer into three eras. During the period from 1760 to 1845 gypsum was used widely, and its beneficial effects were over estimated. This era had its genesis in Europe from the experiments of the Reverend A. Meyers in the Canton of Berne in 1768 (21). During this period, Benjamin Franklin was given credit for introducing gypsum into the United States (111). He is said to have chosen a meadow in Pennsylvania adjacent to a main road near Washington, D. C., and to have written on it with gypsum, the words, "This land has been plastered". Where the gypsum had been applied a lush growth resulted and, thus, the words could be read easily from the road. During the last part of the eighteenth century and the first half of

the nineteenth, gypsum was used extensively in the Eastern United States as a fertilizer and generally increased the yields of certain crops, especially red clover (21). Boussingault was given most of the credit for bringing the first era to an end by proving, as he believed, that the beneficial action of gypsum was due to its lime content, and not to the influence of sulfur. Leibig thought that gypsum absorbed ammonia from the air and that the plants were benefited thereby (3).

For about sixty years following the first era, the need for sulfur additions to the soil was ignored, and the use of the gypsum was discouraged by agricultural scientists. During this period superphosphate, and sulphates of ammonia and potassium were in use as constituents of fertilizer mixtures. The double, triple super, and metaphosphates had not yet appeared. The beneficial effects of sulphur were attributed to an increase in the availability of potassium in soil (3).

The present era started about 1900 in Russia where the need for sulphur fertilization was shown (3). Further work was initiated by three English agronomists, who established field plots in rural areas. Following this work various localities have been found to be deficient in sulfur. The modern period was also enhanced by improvements in the methods of determining the sulfur content of plant material by a succession of chemists from Berthelot in 1888 to Barlow in

1904. The improved methods indicated that crops removed from the soil about as much sulfur as phosphorus. These scientists found that sulfur would have to be added periodically to the soil for continued and increased crop production because of the low quantity and high mobility of this element in the soil. Research on the sulfur problem was started in this country about 1910 by Hart and Peterson (47), and their conclusions were similar to those reached by the early workers.

According to Alway (3), early work to determine the amount of sulfur carried into the soil by precipitation was conducted by Brandes of Germany in 1825, and by Pierre in 1851 and Smith in 1852, both of England. They found that more sulfur was brought down near cities and industrial centers than in rural areas. In America during the early 1900's many errors were present in the determinations of the sulfur content of rainwater.

Crocker (21) reported that, today, little response follows the application of gypsum. He attributed this to a sufficiency of sulfur in the soil which was supplied by applications of commercial fertilizers and manures containing large amounts of this element. A still greater source of sulfur in eastern United States comes from sulfur dioxide which is produced by the combustion of coal. This source of sulfur reaches the crops as sulphate.

Millar (83) classified the states of this country into groups with respect to the sulfur status of the soils.

Sulfur has been shown to be deficient in some Florida soils by Neller et al. (98,99,100), Bledsoe and Blaser (12) and Harris et al. (44). Soils of many other countries of the world also have been shown to be deficient in sulfur (6,20, 37,39,41,51).

Lipman and Conybeare (60) prepared an inventory and balance sheet of the soil nutrients in the United States. In their calculations of the sources of sulfur to the soil, they included fertilizers, precipitation, irrigation, seeds, and plants. Sulfur losses included crop removal, leaching, and erosion. They concluded that the net annual loss of sulfur was nearly five pounds per acre per year. They pointed out that nearly one-fourth of the sulfur additions were by irrigation, which was limited to arid and semi-arid land rich in sulfates. It was estimated that forty-five pounds of sulfur were leached per acre per year. As little as five pounds per acre per year were added in the precipitation in rural areas and one hundred pounds in industrial and population centers.

Additions of sulfur to the soil have been made for purposes other than to correct sulfur deficiencies. Sulfur has been added either as gypsum or as elemental sulfur to alkalai soils as a soil corrective (1,11,62,63).

The early work of Stewart (117), 1920, led to the belief that sulfur was derived in sufficient quanties from the atmosphere for normal plant growth. Kelly and Midgley (57)

indicated that increased growth of plants from the use of gypsum was due to the calcium supplied rather than to the sulfur.

Sulfur in Soils

Forms -- Sulfur is insoluble in water and rather inert. In its elemental form it is of no direct value as a nutrient for crops. An important property of sulfur was found to be the ease with which it is oxidized in the soil (63). MacIntire et al. (68) stated that sulfur reserves in humid soils occur chiefly as organic residues from which sulfates are generated. The organic forms are colloidal and rather easily decomposed. Simple and more available forms exist as sulfate and sulfites of calcium, potassium, and magnesium (61). Conrad (18) reported on the retentive power of soil with regard to sulfur in various compounds, and gave the forms in which the sulfur may have been retained. Sauchelli (109) reported that gypsum furnishes sulfur in the form of sulfate and that it tends to liberate potassium, by reacting with insoluble potassium silicates. Gypsum exchanges its calcium for potassium and magnesium of the clay-humus complex. Evans and Rost (32) reported the various sulfur fractions and ratios in Minnesota soils. Chernozems and black prairie soils have a carbon/nitrogen ratio of about 11.5 and a carbon/organic sulfur ratio of 100. Of the total sulfur, 3.5 per cent is sulfate, 24 per cent inorganic non-sulfate, and the

remaining 72 per cent is organic sulfur. Podzols have a carbon/nitrogen ratio of 13, a carbon/organic sulfur ratio of 225, and a nitrogen/organic sulfur ratio of 17. Of the total sulfur in the podzols, about 9 per cent is sulfate, 44 per cent inorganic non-sulfate, and 47 per cent organic.

Sulfofication.-- Neller (95) reported that sulfur underwent rapid oxidation after mixing with a warm, moist, arable soil and that it oxidized to the trioxide. Most of the oxidation was microbial. Andrews (9) stated that the sulfuric acid combined with lime, if present, and formed calcium sulfate. In this process one pound of sulfur neutralizes three pounds of lime. Neller (93) showed that from thirty to forty per cent of the sulfur applied at rates of 50 to 1,000 pounds per acre in Palouse silt loam was oxidized in the first fifteen days. After longer periods eighty to ninety per cent was oxidized. He also showed that the addition of sulfur up to 1,000 pounds per acre had little effect on the hydrogen ion concentration or on the rate of oxidation of organic matter added to this soil.

Three microbial transformations of sulfur in soils were listed by Starkey (116): (a) oxidation of inorganic sulfur compounds, (b) reduction of inorganic compounds, and (c) transformations of organic sulfur compounds. Numerous organisims were able to perform one or more of these transformations. He showed that the transformation steps of soil organic sulfur were cysteine, cystine, sulfenic acid, sulfinic

acid, sulfite, and finally sulfate. Mosser and Olson (88) showed that Thiobacillus thiooxidans were more numerous than T. thioparus where sulfur had been added to a soil than where none was added. He also showed that the maximum rate of oxidation occured at moisture tensions lower than field capacity.

McKibben (76) reported that sulfur was more readily oxidized in soils low in total sulfur than in those high in total sulfur. Bertramson et al. (11) found that sulfur was of value in correcting manganese deficency in the soils of northern Indiana. Tisdale and Bertramson (130) concluded that an increased manganese avalibility was caused by a reducing effect of sulfur and not by an increase in the acidity. Fraps (33) showed that the oxidation of sulfur had practically no effect on the activity of phosphorus or potassium in soils, but he found that it did increase the permeability of some soils to water. Andrew et al. (8) attributed a beneficial effect of sulfur to an increase of the avalibility of zinc. Martin and Ervin (78) found that a temporary nitrogen deficiency existed where the soil nitrogen was insufficient to meet the requirements of both the plants and the sulfur-oxidizing microorganisms.

Amounts.-- According to Millar (83) a large number of soils of low to medium organic content had a sulfur/carbon ratio of about 1:50 and widened with an increase in organic matter. The total sulfate varied from 2.8 per cent in

chernozems (about 73 per cent in organic form) to 7.9 per cent in podzols (about 45 per cent in organic form). Lyon and Buckman (61) stated that the range of sulfate as SO_3 in the soil is from 0.02 to 0.5 per cent and that representative soils of the humid region had 0.1 per cent SO_3 or 2,000 pounds per acre. McKibben (76) pointed out that the amount of water soluble sulfur in soils was no indication of the total sulfur because soils low in total sulfur may be high in soluble sulfur.

Shedd (113) found that the soils of Kentucky were much lower in sulfur than phosphorus, and stated that sulfur should be in the same class with nitrogen, phosphorus, and potassium with respect to fertilizer practices. Other workers reported the amounts of sulfur in soils from various localities:

Location	Amount of Sulfur	Reference
Alabama	180-302 pounds per acre	(23)
Maryland	460	(23)
Oklahoma	480	(23)
Central States	600	(23)
Illinois (near Chicago)	1220	(23)
Kansas	540	(123)
Kansas	730	(83)
Oregon	300-760	(107)
Oregon	558	(83)
Texas	552	(83)
Wisconsin	400	(47)
Iowa	719-938	(30)
Iowa	815	(83)
Indiana	300-600	(11)
Ohio	1056	(83)
Ohio	400-1,000	(5)
Kentucky	667	(83)
Utah	125-880	(40)
South Africa	20-900 (sandy granite-loam)	(39)

Outgo.-- MacIntire and his co-workers (64,65,66,67, 68,69,70,71,72,73,74) studied the outgo of sulfur for several years. Their work was carried out in lysimeters with special reference to the effect of lime and magnesic materials on sulfate outgo. In 1917 they reported (74) that eight tons of burnt lime per acre reduced the amount of sulfate leached and that thirty-two and one hundred ton rates practically inhibited outward movement of sulfur in solution. In 1923, they reported (72) that the initial depressive effect was not permanent and that seven liming materials at the eight-ton rate substantially increased the loss of sulfates from the surface soil over an eight-year period. They concluded from a five-year study (71), that the use of excessive amounts of either calcium oxide or magnesium oxide was inadvisable in connection with either sulfur or pyrite. In 1925 they demonstrated (67) a typical adsorption phenomena which explained the retention of sulfates by heavily limed soils, based on the ternary systems $CaO-Fe_2O_3-CaSO_4$ and $CaO-Al_2O_3-CaSO_4$. In their 1926 paper (64) they showed that there was no effect of the degree of fineness of the lime nor the depth of incorporation upon the total losses of sulfate from the soil over a four-year period. In 1927 they concluded (65) that, in general, accelerations of sulfate outgo from CaO and CaO-MgO are appreciable and comparable during the first year. In 1938 (66) they obtained sulfate recoveries of 93 to 100 per cent. Complete recoveries occurred only where magnesium and potassium sulfates were

added with dolomite supplements. Neither limestone nor dolomite caused decreases in sulfate outgo. In 1941 (68) their practical conclusions were that sulfates supplied in the form of fertilizers affords current supplies of nutrient sulfur, but such supplies are removed within one year from soils of the humid areas. Acid soils retained small fractions of the sulfate additions. In 1945 (69) they found that potassium metaphosphate either promoted sulfofication or altered the propensity of the soil to retain sulfate, but in 1953 (70) they attributed the effect of limestone and dolomite in the soils to activated sulfofication which resulted in a release of sulfate. The outgo of sulfate from unlimed soil was increased by annual or single incorporations of potassium metaphosphate but not where limestone or dolomite were added.

Plummer (106) found that both large and small amounts of five liming materials increased the solubility of native soil sulfate for a few years. This also was true for small amounts of CaO, but large amounts apparently reduced the loss of sulfur from the soil. Ellett and Hill (28) observed that lime did not give a marked increase in sulfate outgo, but the biological processes which transform sulfur in the soil appeared to be stimulated. When millet and clover were grown the outgo tended to diminish. They also found that the subsoil had a fixing effect because sulfate outgo decreased as the depth of the subsoil was increased. Joffe (53) was in agreement with this when he reported on a seven-year lysimeter

experiment in which leachates were taken from each horizon. He found that 28.4 pounds of sulfur passed through the A_1 horizon, less than one-half this amount through the A_2 horizon, and only a small quantity through the B_1 horizon. The concentrations of sulfur in the leachates differed in that those from the A_2 were two times as great as those from the A_1 and B_1 horizons. He concluded that plants utilize the sulfur from the B_1 horizon.

Thomas et al. (126) found that pH 5.5 was better than pH 7.0 for the growth of alfalfa where sulfur concentrations were low, but there was no pH effect where 90 ppm. of sulfate sulfur existed. Truog (133) stated that at pH 6 and higher, reaction conditions are uniformly favorable for the presence of adequate amounts of available sulfur.

In lysimeter experiments, Volk and Bell (134,135) found that about one-third as much sulfate leached from band placement of gypsum as compared to broadcast, but with more soluble sulfates 52 per cent more sulfate moved from the band placement than from broadcast application.

Relation to Yields and Sulfur Content of Plants.--Gaw and Soong (37) reported elemental sulfur depressed dry weight yields of garden peas. Some workers (14,48,85) found that sulfur additions to the soil caused no increase in yields or increase in the sulfur contents of alfalfa, rape, and clover. Neas (89) observed no differences in yields of tobacco caused by adding sulfur, but the fire-holding capacity

was poorer where sulfur had been added to the soil. Grutz (41) found no yield increases of spinach in dry years or where sulfur was applied at rates less than 450 Kg/ha. Hilder (51) obtained yield responses of clover to sulfur in the field but not in the greenhouse. He attributed this to the release of sulfur from the organic matter caused by soil disturbance. Bledsoe and Blaser (12) found no yield increases of Pensacola Bahia grass. Many investigators (20,48,49,54,84,85,92,104), who observed no yield increases, found increases in the sulfur content in the plants. These plants included barley, rape, alfalfa, and radishes. Other workers (6,11,19,30,44, 51,77,108,113,132,141), who reported only yields, observed increases that were attributed to sulfur additions. Clover, oats, cotton, alfalfa, tobacco, soybeans, turnips, radishes, mustard, and rape were included in their studies. Several other reports (7,12,52,84,94,95,98,99,100,107) showed that both yield and sulfur content of the plants increased where sulfur had been added. The crops included clover, oats, flax, rape, alfalfa, and Pensacola Bahia grass. Alway (2) summed up the responses by stating that when soils contain a certain amount of sulfur the yields are not increased where sulfur is applied, but the percentage of sulfur in the plant is usually increased. Thomas et al. (127,129) supplemented this by reporting that where sulfur is deficient or barely adequate in the soil, practically all of the sulfur in the plants is in the organic form. However, where the supply in the soil is

plentiful, the additional uptake appears in the tissue largely as sulfate, which in extreme cases reaches five to ten times the organic content before it becomes injurious to the system. They found that organic sulfur content ranged between 0.2 and 0.4 per cent. They concluded that the organic fraction was a more significant index to the sulfur needs of the plant than the inorganic fraction.

Legumes generally require larger amounts of sulfur than most non-legumes, except for the Cruciferae. The amount of sulfur removed from the soil by the following crops was reported to be:

Crop	Amount	Sulfur Removed	Reference
Corn	100 bu. per acre	16.0 lbs.	(11)
Wheat	50 bu. per acre	10.2	(11)
Potatoes	400 bu. per acre	32.6	(11)
Alfalfa	4 tons per acre	23.0	(11,30,40)
Tobacco	5,000 lbs. per acre	8.4	(11)
Clover	2 tons	8	(30)
Cabbage		40	(30)
Grains		8-16	(30)
Corn grain	100 bu.	9.7	(40)
Barley grain	100 bu.	7.4	(40)
Oats grain	100 bu.	6.1	(40)
Peas	4 tons	13.6	(40)
Potatoes		5.0	(40)
Sugar beet		13.0	(40)

Effect on Nodulation of Legumes.-- It was found by several workers (7,22,40,94,95,105,107) that sulfur additions to the soil increased nodulation. Gaw and Soong (37) observed that S, $CaSO_4$, Na_2SO_4, $ZnSO_4$, and $FeSO_4$ increased nodulation on peas, but $CuSO_4$, $CrSO_4$, and K_2SO_4 depressed nodulation.

Sulfur in Plants

Metabolism.-- A considerable amount of research has been carried out on the sulfur metabolism of plants (7,25, 26,27,31,54,129,140). Sulfur is an essential element for plants because it is a vital constituent of most plant proteins and their amino acids: cystine, methionine, and glutathione (103,129). The addition of sulfur to soils which were deficient in this element increased the methioine and cystine in plant tissues (49,114,130).

Sulfur requirements of alfalfa varied with the methionine contents of several strains (11). Two of the viatims, thiamine and biotin, found in plants contain sulfur (35,103). It was observed that the sulfur-containing vitamins and amino acids were affected no more severely by sulfur deficiency than were many of the other vitamins and amino acids (91). This indicates that sulfur deficiency affects the general metabolism which in turn affects a number of constituents.

Thomas et al. (129) showed that the absorption of sulfate from the soil and its distribution throughout the plant was rapid; likewise, the reduction of the sulfur and its incorporation into organic compounds was rapid. Radioactive cystine sulfur was isolated from sulfur deficient tomato leaves ten minutes after the addition of radioactive sulfate in nutrient solution. Wood and Barrien (140) found that the

reduction was linked to nitrate-nitrogen reduction and carbohydrate oxidation. Johnson et al. (54) found that manganese influenced the extent of sulfur metabolism.

In view of the essential presence of sulfur in plant proteins, the relation of nitrogen and sulfur is of interest. Nightingale et al. (101), Thomas et al. (128), and Eaton (25) have shown that nitrogen-rich, sulfur-poor plants carried considerable amounts of nitrate, amide, free amino nitrogen, and more than normal amounts of some carbohydrates. They suggested that this condition was due, first, to a lowered rate of nitrate reduction; secondly, to less than normal protein synthesis; and thirdly, to considerable proteolysis in the stem. Evidently sulfur deficiency caused abnormal nitrogen metabolism. When both nitrogen and sulfur were low the abnormalities disappeared, suggesting that there was sufficient sulfur present to allow the plant to utilize all the available nitrogen. In a later paper, Eaton (27) concluded that two opposing factors affect chemical constitution, proteolysis and lessened reductase activity. Ergle and Eaton (31) showed that sulfur deficiency resulted in extra accumulations of phosphorus, calcium, and magnesium, but no changes in iron or potassium.

Deficiency Symptoms.-- Sulfur deficiency symptoms of black mustard are short plants; yellow-green color; small, thin, stiff-textured leaves; and hard stems with a low

moisture content (26). Anthocyanin develops in the leaves and stems under conditions of extreme sulfur deficiency.

Sulfur deficiency in cotton showed fewer bolls but the size, staple length, and lint turn-out were not affected (141). Harris et al. (44) observed that deficiency symptoms were very striking in young plants but almost disappeared as plants approached maturity. Sulfur deficiency symptoms of soybeans were found to be small yellow-green leaves and thin stems. The upper leaves yellow first, the stems are hard and enlongated, and the plants are less succulent (24). Hagler (42) did not observe sulfur deficiency of dewberries grown in sand culture. Chessin (17) found that the length:width ratio of Nicotiana glutinosa leaves was significantly altered by sulfur deficiency. Generally the addition of sulfur to soil delays the maturity of the plants (2, 44, 141). The top growth was affected more than the roots (24, 126). Neller (99) pointed out that in sandy flatwoods soils of Florida sulfur deficiency is more likely to occur than nitrogen deficiency in clover-grass pastures because of the nitrogen fixed by bacteria.

Amounts and Distribution. -- Harrison et al. (45) found by the use of radioactive sulfur that in wheat leaves this element was distributed uniformly in the leaf; however, there was a concentration of sulfur in the kernel at the dough stage. The distribution of sulfur is similar to the distribution of protein. Fried (35) found that the concentration of sulfur in the veins of alfalfa leaves was higher than in

intravenous tissue. Cormack et al. (20) reported that legumes take up higher proportions of fertilizer sulfur than barley. From Beeson's (10) report the sulfur contents of Hubam sweetclover, oat grain, oat straw, and soybeans averaged 0.45, 0.19, 0.25, and 0.29 per cent, respectively. Thomas et al. (127) listed the range of sulfur in alfalfa as 0.38-1.52 per cent. Neller (94) gave sulfur contents of clover as 0.24 and 0.75 where no sulfur and 4,000 pounds per acre were added, respectively. From alfalfa grown on Oregon soils, Miller (84) reported 0.12 and 0.23 per cent sulfur where sulfur was omitted and where it was added to the soil. Hart and Peterson (47) found 0.29 and 0.16 per cent sulfur in alfalfa and red clover. Hall (43) analyzed ten lots of alfalfa and found that the range of sulfur was 0.37-0.56 per cent. Bruce (14) found that the sulfur content of alfalfa on the control plot was 0.44 per cent. Volk et al. (136) observed that liming a soil caused decreases in sulfur content of plant tissues. The sulfur and phosphorus contents of a number of plants reported by Conrad (19), showed that the sulfur/phosphorus ratio ranged from 0.34 for vetch to 2.21 for tobacco.

Sulfur in the Atmosphere

Using radioactive sulfur as a tracer, Fried (35) demonstrated that alfalfa plants took in SO_2 through the leaves and converted it into organic sulfur compounds; thus, he

concluded that SO_2 of the atmosphere was a source of sulfur for the nutrition of plants. Bertramson et al. (11) stated that about six per cent of the crop's requirement could be supplied by absorption from the atmosphere. This was in agreement with the report of Alway et al. (4), who found that not more than 1/16 of the sulfur requirement of alfalfa could be obtained by atmospheric absorption. Frazer (34) observed that plants obtained from localities where there was distinct atmospheric contamination from factories, chimnies, etc. contained several times as much sulfur as corresponding plants from regions where the influence of such atmospheric pollution was negligable.

Several investigations have been conducted on the amounts of sulfur brought down by precipitation. Over a two-year period from 1918-1920 at Ithaca, New York, Wilson (139) found that 26.19 pounds per acre per year were brought down in rainwater. Leland (58) found for the same area between 1931 and 1949 that an average of 48.67 pounds of sulfur per acre per year was brought down in precipitation. Bertramson et al. (11) found that in Indiana the amount brought down varied from 20 to 127 pounds per acre with an average of 27 pounds. MacIntire and Young (75) reported 51.5 pounds per acre at the University Farm at Knoxville, Tennessee, 94.5 pounds per acre in the city proper, and 18.6 pounds per acre seven miles from Knoxville. Alway et al. (4) gave a range

in Minnesota from less than 5 to more than 100 pounds per acre per year. He also showed errors caused by different types of rain collecting devices and gave a method for determining the amount of atmospheric sulfur with lead peroxide candles.

Sulfur in Fertilizers

In 1947 approximately one-third of the total production of sulfuric acid in this country (about 3.5 million tons) was used for the manufacture of commercial fertilizers (110). Sauchelli (109) reported that calcium sulfate occurred as the anhydrate $CaSO_4$, hemi-hydrate $CaSO_4 \cdot 1/2H_2O$, or as dihydrate $CaSO_4 \cdot 2H_2O$ (gypsum). He showed the following chemical reactions as the ones that occurred in the manufacture of superphosphate.

$$Ca_{10}P_6O_{24}F_2 + 10\ H_2SO_4 = 6\ H_3PO_4 + 10\ CaSO_4 + 2\ HF$$

$$Ca_{10}P_6O_{24}F_2 + 14\ H_3PO_4 + 10\ H_2O = 10\ CaH_4(PO_4)_2 \cdot H_2O + 2\ HF$$

Both reactions occur simultaneously. From this he showed that superphosphate is a 50 - 50 mixture of calcium sulfate and phosphatic compounds. Neller (99) further stated that the calcium sulfate is hydrated to gypsum in the soil and that gypsum leaches readily.

Mehring and Bennett (79) stated that more than three-fourths of the tonnage of all fertilizers consumed in 1948 contained more SO_3 than the sum of the tonnages of N, P_2O_5, and K_2O combined. They estimated that the ratio of the

N, P_2O_5, K_2O, and SO_3 contents of all the crops harvested in the United States was 1:0.4:0.6:0.3. Fertilizers high in sulfur are ammonium sulfate, superphosphate, ammonium phosphate (20 per cent P_2O_5, 16 per cent N), and potassium sulfate (79,80). Fertilizers low in sulfur are nitrate of soda, ammonia, ammonium nitrate, calcium nitrate, urea, ammonium phosphate (48 per cent P_2O_5, 11 per cent N), triple superphosphate, muriate of potash (19), and basic slag (79). Millar (83) reported that fertilizer mixtures containing 12-20, 21-40, and over 40 per cent N, P_2O_5, and K_2O contained 8.1, 6.9, and 1.4 per cent sulfur, respectively.

Sulfur in Animals

Gilbert (38) stated that sulfur is not ordinarily thought of in connection with mineral deficiencies of animals because if the diet contains sufficient protein it also contains sufficient sulfur. Painter (103) gave a discussion of the functions of sulfur in the body. He stated that methionine is an essential amino acid; cystine reduces the requirements of methionine, but can not support growth in the absence of methionine; methionine may be converted to cystine in the body; cystine is part of the compound, gluthathione, which may be an intermediate in biological oxidations and reductions; methionine aids in protecting against fatty livers; and the methyl group of methionine serves as a source of labile methyl in metabolism.

Chemical Methods

Turbidimetric Methods for Extractable Sulfur in the Soil. -- The first use of a colorimeter in turbidimetric sulfur determinations was employed by Schreiner and Failyer (112) in 1906. Previous to this, standard candles were used. Brown and Kellogg (13) showed that sulfates could not be extracted from soils by treating with dilute hydrochloric acid because of the interference of organic substances and iron compounds. They further stated that shaking soil with water for eight hours completely extracted sulfates. They recommended the use of a photolometer as a rapid and accurate means of determining sulfates.

Chesnin and Yien (16) proposed a method in which Morgan's (87) soil extracting solution (a 3 per cent acetic acid solution buffered at pH 4.8 with sodium acetate) was shaken for 30 minutes. The ratio of soil to extractant was 1:5. To an aliquot of the extract, 1 gm. of 30-60-mesh barium chloride crystals was added and shaken for 5 minutes. If the sulfur concentration was less than 20 ppm., 1 ml. of a 0.25 per cent solution of gum acacia was added; if 20-40 ppm. sulfur were present, 2 ml. of the gum solution were added. The turbidity was determined within 5 to 30 minutes by a colorimeter using a blue filter. They found that the average difference from the gravimetric method was - 0.74 ppm. sulfur. This was attributed to co-precipitation in the gravimetric

method. No interferences were caused by Ca, Mg, Fe, Al, PO_4, or SiO_3 ions. Ensminger (29) modified Chesnin and Yien's (16) method to test other extractants. He found that about the same amounts of sulfate were extracted by neutral sodium acetate solution, sodium acetate buffered at pH 4.8, KH_2PO_4 solution containing 100 ppm. of phosphorus, and KH_2PO_4 solution containing 500 ppm. of phosphorus. However, 0.1 N HCl solution extracted little or no sulfate. The samples contained only small quantities of water soluble sulfate, except where sulfate had been applied recently.

Smittenberg et al. (115) gave methods for determining different types of sulfur in the soil. They recommended acetone for the determination of extractable sulfur. A rapid test for active sulfides in the soil was proposed by Neekers and Walker (90).

Fractionation of Sulfur in Plants. -- Needham and Hange (91) described a procedure for determining the amount of sulfate sulfur in plant material. The difference between this value and the total sulfur is the organic fraction.

Catalase Activity

According to Lemberg and Legge (59) and Sumner and Somers (122), Thenard observed that animal and plant tissues decomposed hydrogen peroxide. Shonbein, in 1863, considered that all enzymes did this, but it was disproved by Jacobson,

Bourquelot and Loew when they showed that hydrogen peroxide decomposition was caused by a specific enzyme. Loew named this enzyme, catalase, in 1902.

It has been stated that catalase is present in all physiologically active tissue except obligate anerobic microorganisms (86, 121, 122). Taubler (124) showed that catalase prepared from sprouted squash seeds was identical with animal catalase. It seems that little is known about the physiological importance of catalase in tissues. It was reported to be concerned with oxidative metabolism (119). Harrow (46) and others (102, 86, 138) stated that it was apparently present for the purpose of decomposing any H_2O_2 which tended to accumulate in the tissues. Several enzymatic reactions produced H_2O_2 during the course of their activity (46). Sumner and Myrbach (119) stated that catalase cleaves H_2O_2 into O_2 and H_2O but catalyzed no other reactions.

Keilin and Hartree (56) showed that hydrogen peroxide was the only known substance capable of reducing catalase. They also showed that the reduced catalase was readily reoxidized by molecular oxygen. Burris and Little (15) stated that the specifity depended on the protein moietly and on how it is attached to the hematin groups.

Meyer and Anderson (82) and Mitchell (86) stated that the abundance or activity of catalase in tissues was proportional to their oxidative rates or to their metabolic status. Neller (97) found that there appeared to be a definite relation

between the growth or nutritional condition of plants and their catalase activity. His findings (96) on the physiological breakdown of Jonathan apples corroborated the theory that catalase activity measurements may be used as an index of the rate of metabolic activity. Sullivan (118) reported on the effect of hydrogen sulfide on the catalase activity of wheat. She found that hydrogen sulfide caused a slight decrease in the catalase activity of dormant wheat and a considerable decrease in germinating wheat.

Catalase is similar to hemoglobin, both are iron-porphyrin protein-complexes. The only difference is the anion attached to the iron in the porphyrin structure (121). Williams and Beerstrecher (138) stated that catalase contained about 0.1 per cent iron; that the molecular weight was approximately 250,000; and that each molecule contained four hemetin groups. Catalase is extremely active. Mitchell (86) stated that at 0° C. one molecule of catalase decomposed 440,000 molecules of hydrogen peroxide per second. The optimum temperatum was between 0 and 10° C. Sumner and Somers (122) reported that catalase was unstable in acid and it was destroyed at pH 3. The optimum pH was about 7.8. They gave the following partial equations as the ones involved in the decomposition of hydrogen peroxide by catalase.

$$Fe\text{-}OH + H_2O_2 = Fe\text{-}OOH + H_2O$$

$$Fe\text{-}OOH + H_2O_2 = Fe\text{-}OH + O_2 + H_2O$$

Fe-OH represents catalase and Fe-OOH represents catalase peroxide. A definite intermediary complex exists (120). Catalase undergoes only slight deterioration for months, when it is kept in a dark, cold environment, free from micro-organisms (122).

Catalase can be determined quantitatively when its reaction with hydrogen peroxide is carried out in an apparatus in which the rate of evolution of the oxygen can be determined (82). Keilin and Hartree (55) gave the method used to determine the absolute amounts of catalase activity. They used the following equation to calculate the activity:

$$\text{Cat. f} = K/g$$

Cat. f is Kat.f from Katalasefahigkeit (120) or catalase ability, K is reaction velocity, and g is the dry weight of the enzyme preparation as gm/cc.

MATERIALS AND APPARATUS

Greenhouse. -- The greenhouse in which the experiments were carried out parallels a north-south direction. Each spring, usually in April, a water-soluble paint was applied to the entire outer glass surface to reduce the amount of heat inside the greenhouse. Within the greenhouse and parallel to its sides are wooden benches upon which were placed the two-gallon glazed earthenware jars of soil. The water, available for keeping the soil moist, was obtained by demineralizing city water with resin exchange columns. A supply of this water was maintained in two 30-gallon glazed earthenware containers. The water was taken from these in porcelanized buckets and the quart dippers were made of polyethylene. Pipes, from which paint was flaking (Photograph 40), were wrapped to prevent contamination of the soils and special measures were taken to prevent an occasionally trapped bird from roosting overhead.

Equipment for Preparation of Plant Material and Soil Samples. -- In the preparation of plant material, the following items were used; scissors for harvesting plants; and electric, fan-ventilated oven held at approximately 80° C. for drying the plant material; a balance, accurate to 0.1 gm., for

weighing the yields; and a Wiley mill, standard model number 2, for grinding plant material. Seives of 20-mesh were used for screening soil samples.

Laboratory Materials and Apparatus. -- The apparatus used in the determination of catalase activity included a Waring blender, Model F.C.I. 15, and a Warburg apparatus, serial no. 21065, which was equipped with a thermostat and an agitator. The manometers contained Brodie's solution. An electric ignition type Parr peroxide bomb no. 2001 was available for the determination of total sulfur. The soils were extracted in square cornered, 8-ounce, glass bottles by shaking in a "Precision" equipoise shaker at a speed of 250 strokes per minute with an amplitude of two and one-half inches.

All chemicals used were of "chemically pure" grade. A steam-heated still furnished distilled water which was tested periodically for the presence of sulfates. A detergent was used for washing laboratory glassware. Laboratory samples of less than five grams were weighed on an analytical chainomatic balance; for samples of five grams and above, a 200-gram capacity torsion balance was used.

Two instruments were used for the sulfur determinations. The Hellige turbidimeter, equipped with two filters and two cells of 10 and 20 mm. viewing depths, was suitable for measuring sulfur concentrations up to 30 ppm. The other instrument was a Cenco-Sheard-Sanford photolometer, type A, with five cm. cells and an adapter for 1 cm. cells which gives

a range determinable to 40 ppm. of sulfur. A blue filter, 470 mu., was used in the photolometer.

PROCEDURE

Soil Analyses

Extraction and Determination of Soil Sulfur. -- At the beginning of this study several difficulties were encountered in the proposed method for the determination of sulfur which is extractable from the soil. In the present work sulfate sulfur in the extracts was determined by putting a 50 ml. aliquot of the extract in a 75 ml. test tube, adding, by measure, approximately 0.33 gms. of 20 to 30-mesh barium chloride crystals, then shaking by inversion for one minute and after five minutes comparing the amount of turbidity with a set of standards prepared in the same way. Standard solutions were prepared by diluting a 100 ppm. sulfur solution which was made by dissolving 0.5370 gms. of calcium sulfate in the extractant. The volume was made up to one liter. This procedure was found to be inadequate because some soil extracts were dark colored. In an attempt to alleviate this condition, decolorizing carbon was added to various soils which were to be extracted. This proved unsatisfactory inasmuch as added sulfate was not recovered. Other extractants were tested to determine their usefulness in measuring the amounts of sulfate sulfur in the soil.

Ratios of soil to extractant were also studied. Data obtained in this preliminary work are given in Table 1. Soil extracts from Bray's and Morgan's solution, using a 1:2 ratio of soil to extractant, contained sulfate concentrations that were too great to be measured turbidimetrically but extracts obtained with higher ratios showed only a trace of sulfate.

A similar study was made on Ona fine sand (Table 2). Samples were shaken for fifteen, thirty, and forty-five minutes. When Ona fine sand was extracted with water and with Morgan's solution, the sulfate detected increased as the ratio of soil to extractant increased. The Morgan solution extracts using the 1:2 ratio had to be diluted one-half before the determinations could be made. This relationship did not hold when Bray's solution was used, for with it a 1:8 ratio showed less ppm. of sulfate sulfur than either a 1:2 or a 1:4 ratio. The water extracts from Ona fine sand and Orangeburg loamy fine sand gave sulfates in amounts that varied directly with the ratios. Morgan's solution extracted about five times as much sulfate as water.

Since Morgan's solution extracted larger amounts of sulfate than either Bray's or water, it was used to determine the amount of extractable sulfur in the soils in the 1954 greenhouse experiment. A 1:2 ratio of soil to extractant was used instead of 1:8 even though the latter showed somewhat larger amounts of sulfur in the soil. The extracts from the 1:2 ratio contained higher concentrations of sulfate with

TABLE 1

THE AMOUNT OF SULFUR IN ORANGEBURG LOAMY FINE SAND AS DETERMINED BY THREE EXTRACTANTS AND THREE RATIOS OF SOIL TO EXTRACTANT

Ratio Soil:Extractant	Extractant	Extractable Sulfur in the Soil
		ppm.
1:2	Water	10.2
1:4	Water	14.0
1:8	Water	20.6
1:2	Morgan's	35.0
1:4	Morgan's	1.0
1:8	Morgan's	0.0
1:2	Bray's	35.0
1:4	Bray's	48.6
1:8	Bray's	46.8

TABLE 2

THE AMOUNT OF SULFUR IN ONA FINE SAND AS MEASURED TURBIDIMETRICALLY WITH A PHOTOLOMETER USING THREE EXTRACTANTS, RATIOS, AND LENGTH OF SHAKING PERIODS[a]

Ratio Soil:Extractant	Shaking Time	Sulfate Sulfur Extracted from the Soil		
		Water	Morgan's	Bray's
	min.	ppm.	ppm.	ppm.
1:2	15	10.0	55.0	30.2
1:4	15	13.5	61.2	33.0
1:8	15	16.0	90.0	24.5
1:2	30	10.4	55.2	31.8
1:4	30	14.5	66.8	34.8
1:8	30	21.0	92.0	24.5
1:2	45	12.4	59.8	33.9
1:4	45	16.0	68.0	41.5
1:8	45	21.0	96.5	31.0

[a] Averages of duplicate determinations. C.V., 6.5 per cent

less variation between duplicates. The soil and extracting solution were shaken forty-five minutes. Table 3 is a summary of the results obtained from analyses of Leon, Red Bay, and Rutlege soils after they were cropped in 1954. Almost undetectable amounts of sulfate were apparent in the extracts of Leon and Red Bay soils and none was detected in the Rutlege.

In pursuit of a procedure with which reliable data could be obtained, several theories were tested. The first of these was that soluble organic matter was taking the sulfate out of solution in the extracts. To 40 ml. aliquots of the extracts, 10 ml. of 15 per cent hydrogen peroxide were added and brought to boiling and precipitation was attempted in the manner previously described. The hydrogen peroxide treatment of the extracts did not cause precipitation (Table 4). In another experiment a more vigorous oxidation was carried out by evaporating to dryness on the steam bath 40 ml. of an extract in the presence of 10 ml. of concentrated nitric acid. The volume was again made to 40 ml., and 0, 4, and 10 ppm. of sulfate sulfur were added to separate aliquots. The amounts of sulfate sulfur recovered were 0.15, 0.30, and 9.8 ppm. Blanton fine sand extracts were used in this test. In all cases the recoveries were less than the amounts added.

The gravimetric method for the determination of sulfur, as a barium sulfate precipitate, specifies that the

TABLE 3

SULFATE SULFUR CONTENTS OF LEON FINE SAND, RED BAY FINE SANDY LOAM, AND RUTLEGE FINE SAND USING A 1:2 RATIO OF SOIL TO MORGAN'S EXTRACTANT AND PRECIPITATING WITH 0.33 GRAMS OF BARIUM CHLORIDE

Soil Type	Sulfate Sulfur Extracted			
	Amounts of Sulfur Added to the Soils before Cropping lbs/acre			
	0.00	1.86	7.44	29.76
	ppm.	ppm.	ppm.	ppm.
Leon f.s.	0.14	0.26	0.28	0.30
Red Bay f.s.l.	0.02	0.16	0.18	0.08
Rutlege f.s.	0.00	0.00-	0.00-	0.00

TABLE 4

EFFECT OF HYDROGEN PEROXIDE ON THE PRECIPITATION OF SULFATE IN MORGAN'S SOLUTION EXTRACTS FROM GAINESVILLE LOAMY FINE SAND

Amounts of Sulfur Added before Cropping lbs/acre	Sulfur Extracted	
	Untreated	H_2O_2 Treated
	ppm.	ppm.
0.00	0.35	0.10
1.86	0.40	0.00
7.44	0.35	0.00
29.76	0.30	0.25

precipitation should be carried out in acid solution. Hydrochloric acid was added to a Morgan solution extract of the Gainesville loamy fine sand until the methyl orange end-point was reached. Then 3 ml. excess of forty per cent by volume hydrochloric acid was added per 50 ml. of the extract. Known amounts of sulfate were added to and the recovery was determined. The results (Table 5) showed that below 6ppm. of sulfate sulfur incomplete or no apparent precipitation occured.

It had been observed for the standard solution that with decreases in concentration below 1 ppm. sulfate sulfur there was only a small change in the percentage transmission. Assuming that the prevention of precipitation of sulfate sulfur in soil extracts was caused by the same factor that gave this abnormal standard and that if one could be corrected it would also apply to the other, steps in the procedure were modified in several ways. These included acidification, seeding with a barium sulfate suspension, allowing more time for the precipitate to form, and saturating the standard solutions with barium sulfate. The only modification of the procedure which effected the standard curve was the length of time allowed for the precipitate to form. Figure 1 presents this effect graphically and Figure 2 shows that a satisfactory standard curve can be obtained up to 10 ppm. of sulfate sulfur when a thirty minute interval is allowed for precipitation of the barium sulfate.

TABLE 5

EFFECT OF ACIDIFICATION ON PRECIPITATION OF SULFATES IN EXTRACTS FROM GAINESVILLE LOAMY FINE SAND

Concentration of Added Sulfur in the Soil Extracts	Recovery of Sulfur Added
ppm.	ppm.
0	0.0
1	0.0
2	0.0
4	1.2
6	6.2
8	8.2

TABLE 6

SULFUR EXTRACTED BY MORGAN'S SOLUTION FROM THE SOILS USED IN THE GREENHOUSE EXPERIMENTS WITH A THIRTY MINUTE INTERVAL AFTER ADDING BARIUM CHLORIDE

Soil Type	Sulfur Extracted Year of Experiment	
	1953	1954
	ppm.	ppm.
Leon f.s.	0.10	0.00
Red Bay f.s.l.	0.02	0.02
Rutlege f.s.	0.17	0.25
Blanton f.s.	0.00	0.00
Gainesville l.f.s.	0.05	0.00

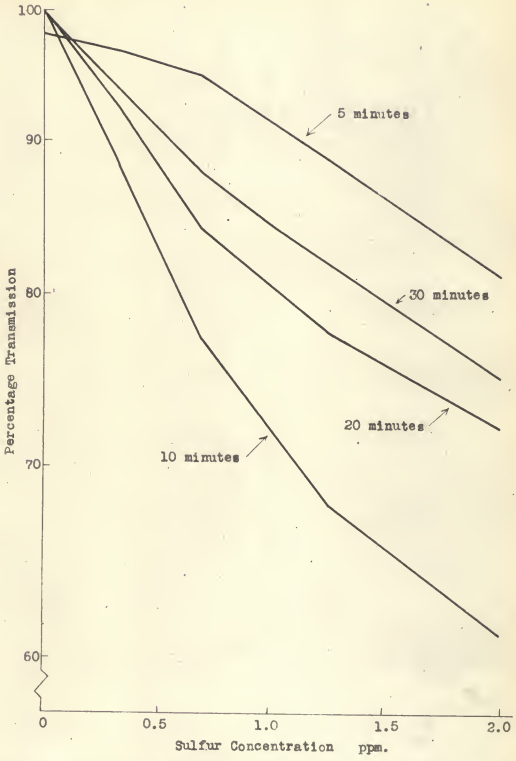

Fig. 1. Effect of the Length of Time on the Formation of Barium Sulfate Precipitate in Standard Solutions.

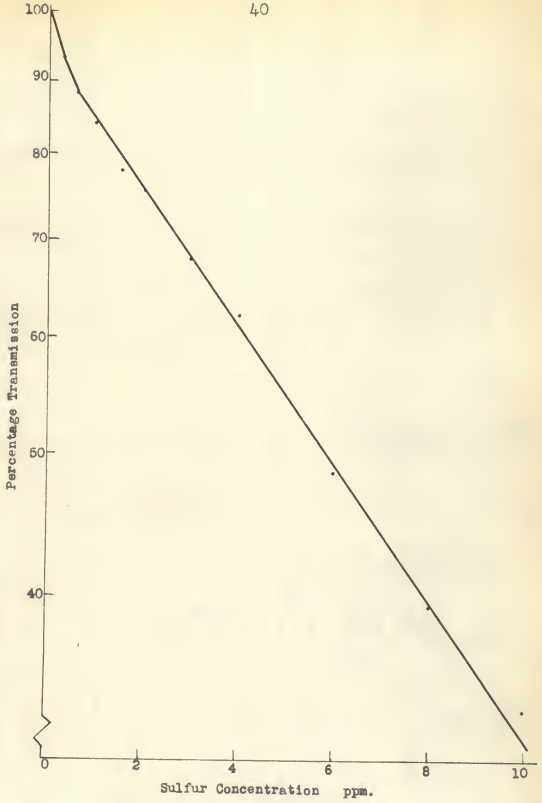

Fig. 2. Standard Curve to 10 Parts Per Million of Sulfur in Morgan's Extracting Solution Allowing a Thirty Minute Interval between Adding Barium Chloride and Determining Percentage Transmission on the Photolometer.

Samples of soils used in the 1953 and 1954 greenhouse experiments before treatment with calcium sulfate, were extracted with Morgan's solution using a 1:2 ratio of soil to extractant; the amount of sulfur in the extracts was determined turbidimetrically allowing a thirty minute period after adding barium chloride. The comparison of the results obtained in this manner (Table 6) with those recorded earlier (Table 3) indicates that the longer time interval for precipitation had no effect on the amount of sulfate sulfur detected in the soil extracts.

All of the determinations heretofore were made on a photolometer using 1 cm. cells. To attain greater precision, samples of Ruston loamy fine sand were extracted and determinations were made using either the turbidimeter or the photolometer with 5 cm. cells. To the extracts that contained less than 1 ppm. of sulfate sulfur, 2 ppm. of sulfate sulfur were added so as to be in the range of sulfur concentrations not covered previously. The extracts with high concentrations of sulfate had to be diluted to be within the range determinable on the photolometer. Sulfate was not recovered in any of the extracts to which it was added (Table 7). Equally important is the fact that when the extracts were diluted by one-tenth, precipitation did not occur. The results showed that in these extracts, sulfate sulfur will not precipitate at concentrations below 2.5 ppm. and that it will precipitate at concentrations above 16 ppm.

TABLE 7

EFFECT OF EXTRACT DILUTION AND OF ADDING KNOWN AMOUNTS OF SULFUR TO SOIL EXTRACTS ON THE DETERMINATION OF SULFATE SULFUR IN RUSTON LOAMY FINE SAND

Block[c]	Depth	Sulfur Added to Extracts	Dilution of Extracts	Sulfur in the Soil	
				Photolometer	Turbidimeter
	Inches	ppm.		ppm.	ppm.
	0-6	2	1.0	a	a
1	6-12	2	1.0	a	a
	12-18	0	1.0	b	41.4
	12-18	0	0.1	1.0	1.0
	0-6	2	1.0	a	a
2	6-12	2	1.0	a	a
	12-18	0	1.0	b	49.0
	12-18	0	0.1	1.1	1.2
	0-6	2	1.0	a	a
3	6-12	2	1.0	b	32.4
	12-18	0	1.0	b	52.6
	12-18	0	0.1	0.0	0.0
	0-6	2	1.0	a	a
4	6-12	2	1.0	a	a
	12-18	0	1.0	b	49.0
	12-18	0	0.1	1.0	1.2

[a] Sulfur added was not recovered. Range in the extracts was 0.1 to 0.5 ppm. sulfur.

[b] Extracts were too turbid at these dilutions for a determination on the photolometer.

[c] From a randomized block experimental design of plots.

Several extracts which were low in sulfate sulfur were composited to determine the critical level below which precipitation would now occur. Sulfate sulfur was added in varying amounts ranging from 0 to 40 ppm. to aliquots of this composite, and then about 0.33 gms. of 20- to 30- mesh barium chloride crystals were added to each solution. At sulfur concentrations of 20 ppm. and above, precipitation was almost spontaneous; at lower concentrations there was no precipitation. After the solutions had set for fifteen hours, 0.33 gms. more of barium chloride were added to each. This caused precipitation of the sulfate added at the rates of 16 and 14 ppm. of sulfur. The addition of more barium chloride to each of the solutions with lower concentrations of sulfur caused precipitation, and in each case the sulfate sulfur added was recovered (Table 8).

This study, relating to extracting and determining sulfate sulfur in the soil, showed that errors occur for the proposed method (16). These errors are present particularly with soils which contain small amounts of sulfate sulfur. The factor causing these errors, whatever it may be, varies with the extracts from different soils.

The present work shows that a definate and critical level of sulfate sulfur in different soil extracts is needed for precipitation to occur. This critical level is reduced by the addition of larger amounts of barium chloride. Thus, there are two ways the proposed method can be modified.

TABLE 8

EFFECT OF SULFUR CONCENTRATIONS IN SOIL EXTRACTS ON THE AMOUNT OF BARIUM CHLORIDE THAT MUST BE ADDED TO BRING ABOUT PRECIPITATION

Sulfur Added	$BaCl_2$ Needed for Precipitation	Sulfur in the Extracts
ppm.	gms.	ppm.
0	10	0.3
1	10	1.9
2	6.0	3.9
4	3.3	5.8
8	1.6	10.8
14	0.66	16.3
16	0.66	17.7
20	0.33	21.5

Known amounts of sulfur can be added to raise the concentration of sulfate sulfur in the extracts above the critical level, or more barium chloride can be added to the extracts. The incorporation of either or both modifications will give results which can be used for relative comparisons of the amounts of sulfate sulfur extractable from a soil.

Accordingly, the sulfate sulfur extracted by Morgan's solution as reported in the following tables was determined by shaking a 1:2 ratio of soil to extractant for 45 minutes, filtering through a Whatman 32 filter paper, taking an aliquot of 40 ml. and adding 10 ml. of a 20 ppm. sulfate sulfur standard. Increments of 0.33 gms. of 20- to 30- mesh barium chloride crystals were added until a precipitate could be seen in the solutions. Concentrations of 4 ppm. sulfate sulfur are readily visible when precipitated in this way and viewed from the bottom of the tube. The turbidity was compared to standards prepared in the same way. The Leon, Red Bay, Rutlege, Blanton, and Gainesville soils required 3.3, 10, 6.0, 6.0, and 3.3 gms. respectively of barium chloride per 40 ml. of extract.

Total Organic and Sulfate Sulfur of Soils. -- The method used for total organic and sulfate sulfur in the soils is basically the official method (8.1) for the determination of total sulfur in plant material. The determination requires a magnesium nitrate solution which is made by dissolving 150 gms. of magnesium oxide in nitric acid, 1:1,

avoiding an excess of acid; adding a little magnesium oxide in excess, boiling, diluting to nearly one liter, filtering, and diluting to exactly one liter. It is important to make the volume to almost one liter before filtering; otherwise, crystals form as the solution cools before the material can be filtered.

A five gram sample of soil was ashed with 2 ml. of the magnesium nitrate solution at 450° C. until the mass was white. After cooling, the contents were heated to dryness two times on the steam bath after adding 20 ml. of 40 per cent hydrochloric acid each time. The heating was continued one hour after dryness. The sample was moistened with 40 per cent hydrochloric acid after which about 20 ml. of water were added, brought to boiling, transferred to 100 ml. volumetric flasks, made to volume and filtered. After appropriate dilutions, the sulfate sulfur content was measured turbidimetrically using approximately 0.33 gms. of the sized barium chloride crystals.

Organic Matter and pH. -- Organic matter content of the soils was determined by the Wakley-Black (137) method, and pH was determined by means of a glass electrode using a Leeds and Northrup potentiometer and a soil-water ratio of 1:2.

Plant Analyses

Catalase Activity. -- The plant material, taken as described later was placed in waxed paper bags and stored temporarily at 10° C. The samples were then cut into approximately quarter-inch lengths, mixed, and a portion equal to the weight of the smallest sample was taken from each sample. This was thoroughly blended with water and an excess of calcium carbonate in a Waring blender. The time required for blending varies with the plant material but ranged from two to five minutes. After trial runs and appropriate dilutions had been made, a 2 ml. aliquot was pipetted into the manometer flask proper of the Warburg apparatus. One ml. of 5 per cent hydrogen peroxide was pipetted into the side-arm of the flask, after which it was allowed to reach a temperature equilibrium with the water bath at 28.5° C. The manometer was adjusted, the stopcock closed, and the hydrogen peroxide added to the plant material by removing and tilting the manometer and flask. The manometers were immediately returned to their positions and shaken at the rate of 134 oscillations per minute with an amplitude of 4.5 cm. After shaking for a definite time interval, a reading was taken. The length of time selected was that necessary for the reaction to be nearly completed. The difference between the original reading and the final reading gave the amount of oxygen liberated or the relative catalase activity of the plant material.

The five manometers used were selected for uniformity; however, since a variation existed among them, the same manometer was used for all treatments within a given soil type so as to reduce errors. The amount of plant material varied from 2 to 40 mgm. for the oats and Hubam harvested April 1, 1954, and the millet harvested May 21, 1954.

Total Sulfur. -- Total sulfur in the plant material from the 1953 experiments was determined by the Parr Bomb method. Total sulfur in the plant material from the 1954 experiment was determined following the procedure in Methods of Analysis of the Association of Official Agricultural Chemists (81) with the exception that the sulfate sulfur in the solutions was determined turbidimetrically, as already described, instead of gravimetrically.

Plant Responses to Native and Added Sulfur in Five Soils - 1953

Experiments were planned to determine whether or not sulfur deficiencies are likely to occur in certain legumes and grasses grown on contrasting soils of Florida.

The soils selected comprise a wide varity of types including Rutlege fine sand, a very poorly drained acid flatwoods soil; Leon fine sand, an imperfectly to poorly drained acid flatwoods soil; Blanton fine sand, shallow phase, a well drained soil developed from thick deposits of unconsolidated fine sands on undulating relief, strongly to very acid and

in close association with the Leon soils; Gainesville loamy fine sand, undulating phase, formed from thin beds of fine sand and fine sandy loam, partly mixed with material developed from underlying limestone that may or may not be phosphatic, and has good drainage (125); and Red Bay fine sandy loam, a well drained soil derived from marine deposits of noncalcareous sands and clay, and characterized by a relatively deep surface overlying a friable sandy clay subsoil (50). The Red Bay fine sandy loam was obtained from the West Florida Experiment Station and the others from Alachua County. The Leon and Red Bay soils had been used the previous year in a greenhouse experiment to study the availability of phosphorus as related to type of phosphate either with or without the addition of gypsum. A uniform application on nitrogen, potassium, and minor elements was added to each soil. Oats was used as the index crop and three harvests were made. The experiment was carried out in the greenhouse in 2-gallon jars in a randomized block design with three replications.

In the preparation of these soils, jars of Leon fine sand which had received gypsum were thoroughly mixed together and returned to the containers. The same procedure was carried out for the jars of soil which had received no gypsum. The Red Bay fine sandy loam was prepared in the same way as the Leon fine sand. The Rutlege, Blanton and Gainesville soils were obtained from the top six inches of the

profile from virgin sites, air dried in the greenhouse, and screened from large roots through a quarter-inch screen. Analyses of the soil before treatment included pH, water soluble sulfate sulfur, sulfate sulfur extractable by Morgan's solution, total organic and sulfate sulfur, percentage of organic matter, moisture equivalent, and texture,

Each 2-gallon jar was weighed in order to aid in the maintainance of a constant moisture relationship in each jar for each soil type. Equal weights of air dry soil were added to eighteen 2-gallon jars for each soil type. The weights of the soil used in the experiments were 8,440, 7,340, 8,720, 8,475, and 7,940 gms. per jar for Leon, Red Bay, Rutlege, Blanton, and Gainesville soils, respectively. These amounts filled the jars to about one and one-half inches from the top.

Precipitated calcium carbonate was mixed with the soil to the full depth of the jars containing Gainesville, Blanton, and Rutlege soils at the rate of one-half, one, and two tons per acre, respectively. Minor elements were added to the soil of each jar on an area basis, calculated as 8.24×10^{-6} acres per jar, at the rate of 10 lbs. per acre of $CuCl_2 \cdot 2H_2O$, $MnCl_2 \cdot 4H_2O$, and $ZnCl_2 \cdot 6H_2O$, and 5 lbs. per acre of $Na_2B_4O_7 \cdot 10H_2O$. Nitrogen, phosphorus and potassium were added to the soil in each jar at a rate equivalent to 1,000 lbs. per acre of 4-12-8 fertilizer. Nitrogen was added in the form of NH_4NO_3 and KNO_3, phosphorus as $Ca(H_2PO_4)_2 \cdot H_2O$, and potassium

as KNO_3. All the nutrients except the lime were added in solution. No minor elements or lime were added to the Leon or Red Bay soils since they had received applications of both the previous year. Top dressings of nitrogen, phosphorus and potassium were applied in solution as needed to prevent their deficiency in the plants. In addition to these, the Rutlege fine sand received a top dressing of magnesium oxide as a deficiency of this element was suspected.

Each soil type was set up as an individual experiment in a randomized block design with three replications. Each block contained six jars which were placed parallel with the bench. The six treatments comprised a factorial of three crops grown on each soil, three of which received an application of gypsum at the rate of 25 lbs. per acre. The other three received no gypsum. The crops grown as indicators were oats, (Avena sativa); pangolagrass, (Digitaria decumbens); and Hubam sweetclover (Melilotus alba). Hairy indigo, (Indigofera hirsuta), was planted following Hubam sweetclover. Dates of the various operations are given in Tables 24 and 25.

Twelve oat seeds were planted per jar and after emergence the plants were thinned to eight. Approximately fifteen Hubam sweetclover seeds were planted per jar and thinned to six plants. Pangolagrass sprigs were obtained from a pasture and six were sprigged per jar. The soils were kept moist by watering daily and by occasionally weighing the

jars of soil so as to maintain the moisture uniformly within each soil type.

Catalase activity was determined in the oats grown on Leon and Red Bay soils thirty-eight and forty-five days after seeding and in pangolagrass twenty-seven days after planting. The oat samples were of the first full leaf from the top of the plants and the replications were combined. The pangolagrass leaves were taken at random along the shoots. The samples of hairy indigo consisted of the first full leaves from the top of the plants. Catalase activity was determined just before the last harvest of pangolagrass and hairy indigo. Replications were analyzed separately.

When insect infestation appeared; the plants, jars, bench, and floor were sprayed with a solution of parathion. Photographs were taken when the plants on some soils showed sulfur deficiency symptoms.

Two harvests were made of the oats grown on Leon and Red Bay soils. The oats were in the vegetative stage for the first harvest and were mature for the second. The second harvest was divided into grain and straw. One cutting was made of the oats grown on the other soils and it was also divided into samples of grain and straw except on Rutlege fine sand where the oats did not mature. A composite of two cuttings of vegetative Hubam and pangolagrass comprised the first harvest on Leon and Red Bay soils but only one cutting was made on the other soils for this harvest. After the

harvest, the soils where Hubam had grown were cultivated lightly and planted with hairy indigo. The second harvest of pangolagrass represents a composite of three cuttings which were made each time the longest shoots had attained a length of approximately three feet. Hairy indigo was in the pre-bloom stage at the time of harvest. The harvested plants were allowed to air dry in the greenhouse after which the material was oven dried at approximately 80° C. for eight to ten hours and then removed from the oven and allowed to equilibrate with the moisture in the air. A study of the equilibrium of oven-dry, unground plant material with atmospheric moisture was made on six samples each of hairy indigo and pangolagrass. This showed that after thirty and forty-five minutes, respectively, there was no significant gain in weight.

The yields of the harvests were obtained and the material was ground to pass through a 2-mm. seive. Total sulfur was determined on one gram samples of the material by the Parr Bomb method where the barium sulfate precipitate is measured gravimetrically.

The soils in the jars were sampled by combining ten plugs per jar taken with a sampling tube. The samples were air-dried, screened through a 20-mesh sieve, and stored in pint ice-cream cartons. The soils in the jars were covered with paper during the fallow period between these experiments and the 1954 experiment. Sulfur extractable by 0.1 N

hydrochloric acid was determined on the soil samples by shaking a 1:4 ratio of the soil and acid for thirty minutes, filtering through a Whatman 30 filter paper and measuring the amount of sulfur turbidimetrically.

Plant Responses to Sulfur Added at Four Rates to Five Types of Soil - 1954

In order to determine the effect on plants of levels of sulfur in various soils, a greenhouse experiment was planned which included legumes and grasses, five types of soil and four rates of sulfur applications. A split-split plot design was used with two replications. The main plots were crops. Hubam sweetclover and oats were used for the first planting and soybeans, (Glycine max), and millet, (Setaria italica), for the second. These were randomized within each replication. The twenty jars used for each crop in each replication were divided into sets of four. The jars in each group were filled with a different type of soil and each set of four was placed at random within the crop and parallel with the bench. These were the sub plots. The four jars for each type of soil in each crop and replication were used for rates of sulfur added to the soil. The sulfur was applied as gypsum at the rates of 0, 10, 40, and 160 lbs. per acre which is equivalent to 0, 1.86, 7.44, and 29.76 lbs. of sulfur per acre. Rates represent sub-sub plots. A clearer concept of the plan of this experiment is presented in

Figure 3 and dates of the various operations are listed in Table 26. The soils used were those from the 1953 greenhouse experiment and were prepared by thoroughly mixing together the soil from the 18 jars of each type of soil and then putting an equal amount, by weight, in each of 16 jars. The weights of Leon, Red Bay, Rutlege, Blanton, and Gainesville soil per jar were 8,500, 7,300, 6,000, 8,900, and 8,000 gms., respectively. Samples were taken at this time for later analysis which included water soluble sulfate sulfur, sulfate sulfur extracted by Morgan's solution, total organic and sulfate sulfur, percentage of organic matter, pH, exchangable K_2O and CaO, soluble P_2O_5, and soluble nitrogen.

Precipitated calcium carbonate was thoroughly mixed with the Leon and Red Bay soils at the rate of 1,000 lbs. per acre and the soils were kept moist following this addition. A uniform application of nutrients equivalent to 2,000 lbs. of 6-6-6 fertilizer and 6 lbs. of magnesium was added per acre as an initial treatment to each jar on the calculated area basis. The nutrients were added in solution in the form of KNO_3, KH_2PO_4, NH_4NO_3, $MgCl_2 \cdot 6H_2O$ and $CaSO_4 \cdot 2H_2O$. Top dressings of nitrogen, phosphorus, and potassium were made when necessary. Ten oat seeds were planted in each of half of the jars and were thinned to six after emergence. Hubam was planted in the remaining jars at the rate of sixteen to twenty seeds per jar and were thinned to eight plants after germination.

Circles represent jars.

Jar numbers are at the top.

Bottom numbers refer to rates of sulfur added as gypsum:

1 - none

2 - 1.86 lbs. of sulfur per acre

3 - 7.44

4 - 29.76

Soil types are indicated by letters:

L - Leon fine sand

RB - Red Bay fine sandy loam

R - Rutlege fine sand

B - Blanton fine sand

G - Gainesville loamy fine sand

Roman numerals refer to replications.

Legumes, Hubam sweetclover followed by soybeans

Grasses, oats followed by millet

Crops are main plots.

Soil types are sub plots.

Rates of sulfur are sub-sub plots.

Fig. 3. Plan of Greenhouse Rate of Sulfur Experiment - 1954

The first sample of oats used for the determination of catalase activity was taken from the two bottom leaves of each plant. The next sample was obtained from the first full leaf from the top of each plant. The last sampling included both oats and Hubam. The oats samples were taken again from the first full leaf from the top and the Hubam as the fifth set of leaves from the top of each plant.

Photographs were taken at the time of each harvest. The first harvest consisted of vegetative oats. The second was oats in the milk stage and Hubam in the full-bloom stage. The plants from this harvest and from the soybeans and millet were cut, dried, weighed, ground, and stored in the same way as the plants from the 1953 greenhouse experiment except that the material was ground to pass through a 1-mm. sieve. After this harvest, the soil in each jar was thoroughly mixed and soybeans and millet were planted where Hubam and oats had grown. The soybeans germinated poorly, so they were replanted using spergon and arosan treated seeds; six plants were grown in each jar. Approximately thirty millet seeds were planted per jar and were thinned to eight after the plants emerged. After three weeks of growth, a uniform application of nutrients equivalent to 1,000 lbs. per acre of 6-6-6 fertilizer was applied in solution to the soil in each jar. The soybeans and millet which were growing on the Rutlege fine sand were sprayed with a suspension of a colloidal manganese compound two weeks before harvests because a manganese deficiency

developed in these plants.

Before harvesting the plants, their catalase activity was measured. Two millet leaves and one soybean leaf from the upper part of each plant was taken for these determinations. The millet was in the vegetative stage and soybeans were in the pre-bloom stage when they were harvested.

The growth of roots was measured by screening them from the soil of each jar with a quarter-inch sieve, drying, and then shaking on a 20-mesh sieve. This material was weighed for yields and was ground for analysis. Total sulfur was determined in the tops of each harvest and in the roots. After screening the roots from the soil, pint samples were taken by drawing 50 ml. portions at random from each jar. These samples were prepared for analysis in the manner previously described and were analyzed for water soluble sulfate sulfur, sulfate sulfur extractable by Morgan's solution, total organic and sulfate sulfur, percentage of organic matter, and pH.

Correlation of Plant Responses and Chemical Analyses of Soil Profiles - 1955

A greenhouse experiment was planned which involved growing plants on sections of virgin profiles of Gainesville loamy fine sand and Blanton fine sand. These profile samples were taken at the same location as the surface soils which were used in the previous experiments. The surface six inches comprised the first depth of the profile and the next

four and five depths of Blanton and Gainesville soils were twelve-inch sections. The experimental design was a randomized block with three replications using the five sections of the Gainesville loamy fine sand and the four sections of the Blanton fine sand profiles as treatments. Two-gallon jars were used to contain the soil. For the log of operations, see Table 27.

Precipitated calcium carbonate was mixed with the soil in each jar at the rate of 1,000 lbs. per acre. Nutrients equivalent to 1,000 lbs. per acre of 4-12-8 fertilizer were added as $Ca(H_2PO_4)_2 \cdot H_2O$, KNO_3 and NH_4NO_3. Minor elements were applied at the same rate and in the same form as were applied to the virgin soils in the 1953 greenhouse experiments. The soils were kept moist after addition of lime.

Oats were used as the first indicator crop and six plants were grown in each jar. The procedures of watering, top dressing, harvesting, and handling the plant material were the same as those employed in the previous experiment. After harvesting the oats in the vegetative stage, spergon treated soybean seeds were planted at the rate of nine per jar and thinned to five after emergence. When the soybeans were in the early-bloom stage, the plants were harvested, and analyses were made for total sulfur. Photographs were taken at the time of each harvest.

RESULTS AND DISCUSSION

Criteria Used for Measuring Plant Responses

The catalase activity in the plants was measured to indicate the possible presence of sulfur deficiency before the appearance of visible deficiency symptoms. Sulfur deficiency affects the general metabolism of plants and catalase activity is an index of their metabolic activity. Catalase activity was used also to show the relative metabolic activity in the plants at the time of harvest. This gave information as to the effect sulfur additions had on the plants at the time of harvest. This differs from yields which measure the accumulative response over the entire growth period.

Visible differences and deficiency symptoms were recorded when sulfur deficiencies appeared in the plants. Photographs were taken to record the nature of these deficiencies.

Growth weights were obtained since one of the ultimate objectives of this research is to make it possible for producers to obtain higher tonnage yields. However, quantity

is not always the most important objective in crop production. Protein is probably considered mostly in the quality of crops and sulfur deficient crops are usually low in protein. Total sulfur in the plant material, in addition to yields, made it possible to estimate sulfur uptake by plants.

Soluble sulfate determinations were made because it is this form which is most likely to be available to plants. The data can be used also for estimating critical levels of available sulfur in the soils. The total organic and sulfate sulfur determinations were made to give information on the amount of reserve sulfur in the soil. The organic matter analyses help to show whether or not sulfur additions and cropping tends to oxidize the organic matter more rapidly, thus making the native sulfur more available.

The "F" and least significant difference, L.S.D., values were determined to test differences for significance. The terms significant and highly significant refers to differences at the 5 and 1 per cent levels of probability, respectively.

Plant Responses to Native and Added Sulfur in Five Soils - 1953

The results from these experiments show variation in responses to sulfur applications with respect to soil types and crops. Table 9 contains reference data of the soils as to the amounts of extractable sulfur, total sulfur, and

TABLE 9

ANALYSES OF THE SOILS BEFORE ADDITION OF CALCIUM SULFATE
1953

	Soil Type				
	Leon	Red Bay	Rutlege	Blanton	Gaines-ville
	f.s.	f.s.l.	f.s.	f.s.	l.f.s.
Sulfur extracted by Morgan's solution ppm.	13.56	11.58	18.90	2.22	10.67
Water extractable sulfur ppm	5.53	0.77	11.80	1.63	1.97
Total organic and sulfate sulfur ppm.	74.0	143.0	229.6	38.3	85.0
Organic matter per cent	2.66	4.84	6.92	1.68	2.10
pH values	5.54	5.35	6.10	5.22	5.80
Moisture equivalent . . .	6.1	17.0	5.5	3.8	9.9
Mechanical Analysis	(36)				
Water	...	...	2.0	0.2	0.6
Very coarse sand	0.1	0.3	0.9	0.6	1.2
Coarse sand	2.1	3.5	2.8	2.9	3.5
Medium sand	8.5	18.1	23.8	25.5	20.4
Fine sand	55.7	26.8	45.4	55.3	52.4
Very fine sand	27.4	15.9	10.1	8.9	9.1
Coarse silt	4.7	21.2	11.3	3.7	6.3
Fine silt	0.4	3.9	1.9	0.6	1.4
Clay	1.0	10.3	3.7	2.5	5.7

organic matter and pH values, moisture equivalent, and mechanical analyses. The textural grades of Leon fine sand were taken from the source quoted but other data were obtained from samples of the soils which were used.

There were no significant differences in yields from the first harvest of oats (Photograph 1),* Hubam sweetclover, or pangolagrass grown on Leon fine sand where 0 and 4.65 lbs. per acre of sulfur were applied but the sulfur contents were increased at the .01 level of probability by the addition of sulfur (Table 10). Yields were not affected by the addition of sulfur to the soil as there was a sufficient amount of native available sulfur during most of the growth period; however, the oats and pangolagrass showed definite sulfur deficiency symptoms at the time of the first harvest. Photograph 2 shows the pangolagrass before the second clipping of the first harvest. Each harvest consisted of several clippings. The first harvest was a composite of two clippings, made four weeks apart. Yield weights were not taken for each clipping but there was probably a true yield difference at the time of the second clipping. Highly significant yield increases in oat grain and pangolagrass (Photograph 3) were caused by the addition of sulfur to the soil and significant increases in yields of oat straw and of hairy indigo were obtained. The sulfur content of pangolagrass, hairy indigo,

*Photographs are presented in the appendix.

TABLE 10

EFFECT OF NATIVE AND ADDED SULFUR ON PLANT RESPONSES AND ANALYSES OF LEON FINE SAND - AVERAGES OF THREE REPLICATIONS

	Amounts of Sulfur Added lbs/acre	Crop				C.V. per cent
		Oats		Hubam, Hairy Indigo[a]	Pangola-grass	
		Grain	Straw			
PLANT RESPONSES						
Catalase Activity[b]						
March 5, 1953	0	6.2		....	1.7	....
	4.65	6.8		....	1.6	
June 15, 1953	0	....		5.8	6.4	
	4.65	....		7.0	15.0	50.0
First Harvest						
Yields gms/jar	0	14.8		8.8	19.9	
	4.65	14.9		6.9	19.2	12.5
Percentages of	0	0.205		0.204	0.165	
sulfur	4.65	0.444**		0.333**	0.286**	7.9
Second Harvest						
Yields gms/jar	0	6.5	19.7	8.9	25.4	11.7[c]
	4.65	15.5**	25.4*	14.9*	58.4**	14.0[d]
Percentages of	0	0.123	0.043	0.122	0.060	13.3[c]
sulfur	4.65	0.174**	0.094**	0.207**	0.161**	10.6[d]
SOIL ANALYSES						
Total organic and	0	60.9		62.3	65.8	
sulfate sulfur ppm	4.65	65.8		65.7	66.5	9.8
Dilute HCl soluble	0	0.62		0.58	0.27	
sulfur ppm.	4.65	4.10**		7.10**	0.40	29.0
Percentages of	0	2.60		2.70	2.67	
organic matter	4.65	2.69		2.79	2.82	4.2
pH values	0	5.03		5.05	5.08	
	4.65	5.20		5.00	5.19	2.6

[a] Hubam for the first harvest, hairy indigo for the second
[b] Arbitrary units of O_2 produced
[c] For hairy indigo and pangolagrass
[d] For grain and straw
* Different at the five per cent level of probability
** Different at the one per cent level of probability

and both grain and straw of oats were increased at the .01 level of probability.

Grain yields of oats were increased more than straw because of the larger percentage of sulfur in the grain than in the straw which would cause a deficiency to be more severe in the grain. Catalase activity in the pangolagrass seemed to be indicative of the growth rate at the time of the second harvest, but this was not significantly so. Pangolagrass yields were increased more than two fold for the second harvest where sulfur was added. The plants which grew on the soil receiving no sulfur were extremely low in this element. The pangolagrass where sulfur was added may have been in the deficient range since the amount of sulfur extracted from the soil was low and was not significantly higher than that extracted where sulfur was not added.

The highly significant differences in the amounts of sulfur extracted from the soils where 0 and 4.65 lbs. per acre of sulfur were applied and where oats and legumes were grown (Table 10) indicate that there were probably no deficiencies in the plants which were grown where sulfur was added to the soil. The addition of sulfur caused no differences in total sulfur, percentage of organic matter, or pH values of the soil.

Plant responses on Red Bay fine sandy loam (Table 11) were quite variable. The sulfur contents of oats for the first harvest and both the oat grain and straw for the second

TABLE 11

EFFECT OF NATIVE AND ADDED SULFUR ON PLANT RESPONSES AND ANALYSES OF RED BAY FINE SANDY LOAM - AVERAGES OF THREE REPLICATIONS

	Amounts of Sulfur Added lbs/acre	Crop				C.V. per cent
		Oats		Hubam, Hairy Indigo[a]	Pangola-grass	
		Grain	Straw			
PLANT RESPONSES						
Catalase Activity[b]						
March 5, 1953	0		12.9	....	13.0	
	4.65		13.0	....	11.0	....
June 15 1953	0		....	9.8	15.0	....
	4.65		....	11.0	6.5**	24.6
First Harvest						
Yields gms/jar	0		14.4	5.9	22.0	
	4.65		14.1	9.9*	18.1*	16.1
Percentages of sulfur	0		0.262	0.281	0.208	
	4.65		0.358**	0.328**	0.250**	4.5
Second Harvest						
Yields gms/jar	0	26.3	30.9	27.2	63.9	16.2[c]
	4.65	25.4	30.8	20.9	64.3	4.1[d]
Percentages of sulfur	0	0.147	0.071	0.158	0.120	8.3[c]
	4.65	0.184**	0.189**	0.180	0.208**	6.8[d]
SOIL ANALYSES						
Total organic and sulfate sulfur ppm	0		120	123	109	
	4.65		156**	138	143**	7.2
Dilute HCl soluble sulfur ppm.	0		0.40	0.22	0.27	
	4.65		3.33**	1.56**	0.13	36.8
Percentages of organic matter	0		4.60	4.61	4.53	
	4.65		4.51	4.46**	4.49	1.3
pH values	0		5.22	5.03	5.23	
	4.65		5.10	4.88	5.06	2.2

[a] Hubam for the first harvest, hairy indigo for the second
[b] Arbitrary units of O_2 produced
[c] For hairy indigo and pangolagrass
[d] For grain and straw
* Different at the five per cent level of probability
** Different at the one per cent level of probability

harvest were increased at the .01 level by the addition of sulfur to the soil but there were no yield differences (Photograph 4). This is caused by an uptake of sulfur in amounts larger than necessary for normal growth and a conversion of some of the organic sulfur into available form. The absence of differences in yield and sulfur content of the hairy indigo indicates that the added sulfur had been taken up by the previous crop of Hubam leaving only a small amount of residual sulfur for the hairy indigo. The significant decreases in yield and highly significant increase in percentage of sulfur in the first harvest of pangolagrass where sulfur was added to the soil could have been caused by increased microbial activity in the soil and a subsequent competition between the plants and the microorganisms for nutrients other than sulfur. There were no yield differences in the second harvest of pangolagrass (Photograph 5) but the sulfur content was higher at the .01 level of probability where sulfur was added to the soil. The reason for the highly significant decreased catalase activity in pangolagrass at the second harvest where sulfur was added is not clear.

The addition of sulfur to Rutlege fine sand caused no differences in plant responses or soil analyses (Table 12). The Rutlege fine sand was unknowingly obtained at a location which was influenced by limestone and was not normal for a virgin site. Assuming that the reaction of the soil was

TABLE 12

EFFECT OF NATIVE AND ADDED SULFUR ON PLANT RESPONSES AND ANALYSES OF RUTLEGE FINE SAND - AVERAGES OF THREE REPLICATIONS

	Amounts of Sulfur Added lbs/acre	Crop			C.V. per cent
		Oats	Hubam, Hairy Indigo[a]	Pangola-grass	
PLANT RESPONSES					
Catalase Activity[b]					
June 15, 1953	0	....	9.2	20.3	
	4.65	....	7.9	19.4	28.1
First Harvest					
Yields gms/jar	0	18.0	7.5	13.1	
	4.65	17.1	8.2	12.5	26.2
Percentages of sulfur	0	0.202	0.308	0.169	
	4.65	0.227	0.299	0.183	16.1
Second Harvest					
Yields gms/jar	0	....	4.1	42.8	
	4.65	....	3.7	43.3	12.6
Percentages of sulfur	0	....	0.236	0.106	
	4.65	....	0.232	0.123	8.0
SOIL ANALYSES					
Total organic and sulfate sulfur ppm	0	237	254	236	
	4.65	231	239	247	33.0
Dilute HCl soluble sulfur ppm.	0	2.60	(c)	0.22	
	4.65	2.53	1.29	(c)	34.2
pH values	0	6.80	6.81	6.81	
	4.65	6.73	6.73	6.98	2.6

[a] Hubam for the first harvest, hairy indigo for the second
[b] Arbitrary units of O_2 produced
[c] None detected
No significant differences

that of a typical virgin Rutlege fine sand, lime was added at the rate of two tons per acre. The resulting pH was about 6.8 and caused lime induced minor element deficiencies in the plants which showed symptoms of spotted, pale-green color, stunted growth, coagulated chlorophyll, curled leaves, and loss of turgidity. Sulfur deficiency may have been complexed with other deficiencies. Magnesium oxide was added to the soil because a deficiency of this element was suspected. Beneficial effects were not caused by this application.

Small amounts of native total and extractable sulfur were present in Blanton fine sand (Table 9). As a consequence of this there were no general large plant responses caused by the addition of a small amount of sulfur to this soil (Table 13). Oat grain yields were significantly greater at the .01 level where sulfur was added, but there were no differences in yields of straw or percentages of sulfur in either grain or straw caused by sulfur addition. Six weeks after planting the oats there were slight deficiency symptoms (Photograph 6) but no apparent yield differences at that time. Hubam gave no differences in yield but a larger percentage of sulfur at the .01 level was present in the plants grown on the soil which received a sulfur application. At the time of harvest no deficiency symptoms had appeared which indicates that the plants growing where sulfur additions were omitted were not in the deficient range even though their sulfur content was lower than the plants where sulfur was added to

TABLE 13

EFFECT OF NATIVE AND ADDED SULFUR ON PLANT RESPONSES AND ANALYSES OF BLANTON FINE SAND - AVERAGES OF THREE REPLICATIONS

	Amounts of Sulfur Added lbs/acre	Crop				C.V. per cent
		Oats		Hubam, Hairy Indigo[a]	Pangola-grass	
		Grain	Straw			
PLANT RESPONSES						
Catalase Activity[b] June 15, 1953	0	....		9.7	11.1	
	4.65	....		9.4	10.1	27.6
First Harvest						
Yields gms/jar	0	11.5	18.8	5.4	9.7	7.6[c]
	4.65	16.2**	21.0	5.9	10.2	5.7[d]
Percentages of sulfur	0	0.092	0.047	0.161	0.079	13.7[c]
	4.65	0.099	0.043	0.230**	0.115*	7.9[d]
Second Harvest						
Yields gms/jar	0	....		14.5	22.3	
	4.65	....		20.7*	23.1	15.4
Percentages of sulfur	0	....		0.068	0.066	
	4.65	....		0.072	0.058	12.9
SOIL ANALYSES						
Total organic and sulfate sulfur ppm	0	50.2		44.6	38.9	
	4.65	39.7**		40.1	40.1	11.5
Dilute HCl soluble sulfur ppm.	0	0.87		0.49	0.80	
	4.65	0.66		0.58	0.27	23.3
Percentages of organic matter	0	1.58		1.61	1.59	
	4.65	1.58		1.61	1.55	4.0
pH values	0	5.97		6.03	6.04	
	4.65	5.93		5.93	5.90	2.3

[a] Hubam for the first harvest, hairy indigo for the second
[b] Arbitrary units of O_2 produced
[c] For Hubam and pangolagrass
[d] For grain and straw
* Different at the five per cent level of probability
** Different at the one per cent level of probability

the soil. The increased yields (Photograph 7) of hairy indigo receiving sulfur and the magnitude and similarity of the sulfur contents of all the plants indicate that at the time of harvest all plants were deficient in sulfur. Catalase activity measurements indicated similar conclusions. Six weeks after planting, pangolagrass exhibited extreme deficiency symptoms. In addition to the usual symptoms, the plants growing on the soil to which sulfur was not applied had a pale red color and the tips of some of the leaves were dying (Photograph 8).

The responses of plants caused by the addition of sulfur to Gainesville loamy fine sand (Table 14) were quite marked in some cases but not in others. Six weeks after planting oats, definite but slight deficiency symptoms appeared (Photograph 9), but no yield differences were apparent at that time; however, at the time of harvest, which was thirteen weeks after planting, there was a highly significant increase in yields of oats straw and a significant increase in percentage of sulfur in the oat grain which could be attributed to the addition of sulfur to the soil. These responses could be explained by an accumulation of sulfur in the vegetative growth of oats in the early part of the growth period which showed up as a yield response, but as the plants matured the accumulation of sulfur was manifest in the grain. Hubam showed no yield response to sulfur after six weeks of growth (Photograph 10), but there was an increase in

TABLE 14

EFFECT OF NATIVE AND ADDED SULFUR ON PLANT RESPONSES AND ANALYSES OF GAINESVILLE LOAMY FINE SAND - AVERAGES OF THREE REPLICATIONS

	Amounts of Sulfur Added lbs/acre	Crop				C.V. per cent
		Oats		Hubam, Hairy Indigo[a]	Pangola-grass	
		Grain	Straw			
PLANT RESPONSES						
Catalase Activity[b]						
June 15, 1953	0	....		30.4	11.4	
	4.65	....		33.3	11.4	12.8
First Harvest						
Yields gms/jar	0	12.0	26.2	9.7	15.0	10.5[c]
	4.65	13.8	31.9**	9.2	17.6	4.3[d]
Percentages of sulfur	0	0.097	0.043	0.162	0.085	15.8[c]
	4.65	0.113*	0.047	0.218*	0.130*	7.6[d]
Second Harvest						
Yields gms/jar	0	....		20.9	27.9	
	4.65	....		26.7**	32.8**	5.2
Percentages of sulfur	0	....		0.057	0.057	
	4.65	....		0.073*	0.084**	8.7
SOIL ANALYSES						
Total organic and sulfate sulfur ppm	0	61.8		59.7	60.8	
	4.65	61.2		61.1	60.9	5.9
Dilute HCl soluble sulfur ppm.	0	1.29		(e)	0.13	
	4.65	1.29		0.36	0.13	58.5
Percentages of organic matter	0	1.67		1.62	1.62	
	4.65	1.69		1.63	1.64	2.0
pH values	0	6.03		6.00	6.04	
	4.65	6.03		6.06	6.04	1.1

[a] Hubam for the first harvest, hairy indigo for the second
[b] Arbitrary units of O_2 produced
[c] For Hubam and pangolagrass
[d] For grain and straw
[e] None detected
* Different at the five per cent level of probability
** Different at the one per cent level of probability

percentage of sulfur in the plants at harvest time, which was nine weeks after planting, where sulfur had been added to the soil. Evidently there was enough native sulfur to support the small amount of growth whether sulfur was added or omitted. The increased yields and sulfur contents of hairy indigo (Photograph 11) show that the amount of native available sulfur had been decreased sufficiently to bring about an increase in yields where sulfur had been added. The sulfur deficiency symptoms in the pangolagrass were about the same for the plants grown on Blanton fine sand as for those grown on Gainesville loamy fine sand. Photograph 12 again shows the dying back of some of the leaf tips. Although there were no differences in yields, there was an increase in the sulfur content of the plants at the first harvest. Increased yields at the .01 per cent level and sulfur contents of the pangolagrass in the second harvest resulted from the addition of sulfur to the soil. Photograph 13 shows the pangolagrass at the time of the third cutting of the second harvest. These differences were caused by increased growth and uptake in the first part of the growth period because at harvest time there were no differences in the metabolic activity of the plants. This was indicated by catalase determinations.

Plant Responses to Sulfur Added at Four Rates to Five Types of Soil - 1954

The data obtained in this experiment were analyzed as split plots or split-split plots depending on whether the data consisted of one crop or two crops respectively except for the catalase activity determinations in soybeans and millet for which only one replication was sampled. The data consisting of one crop, as was the case with oats, where analyzed statistically denoting soil types as main plots and rates of sulfur additions as sub plots. However, when the data were classified into crops, the crops were designated as main plots, soil types as sub plots, and rates of sulfur additions as sub-sub plots. In the analysis of the catalase activity in soybeans and millet, crops were used as replications, soils as main plots, and rates of sulfur applications as sub plots.

The data presented in Tables 16 through 20 are averages of two replications, with the exception mentioned above, and include least significant differences at the 5 and 1 per cent level of probability, where applicable, for comparison of the values obtained where sulfur was added to the soil at the rate of 0, 1.86, 7.44, and 29.76 lbs. per acre. The coefficients of variation listed in Table 28 indicate the relative variability of the data which were analyzed statistically as described above. Regression analyses of plant responses on Leon, Blanton and Gainesville soils were

carried out to examine the polynomial trends of the plant responses on rates of sulfur additions. Table 29 gives the variats, degrees of freedom, and symbols used in these analyses.

The conformity of the response trends to linearity was calculated and the slopes of the lines were determined. These regression analyses and the slopes of the responses, "b" values, are given in Tables 30, 31, and 32.

Table 15 contains reference data obtained from the analyses of the soils used in the level of soil sulfur greenhouse experiment in 1954 as to the amounts of extractable and total sulfur, organic matter, and major nutrients and pH. A comparison of these values with those in Table 9 shows generally more sulfur was present in the soil at the beginning of this experiment than was present at the beginning of the previous year experiments.

Plant responses and soil analyses on Leon fine sand (Table 16) appeared as significant effects caused by the addition of sulfur to the soil at four rates. Generally significant responses appeared as increases caused by the addition of sulfur at higher rates. There are a few exceptions particularly between the 0 and 1.86 lbs. per acre of sulfur additions with respect to sulfur concentrations in the plants. Figure 4 is a graphical presentation of the significant plant responses with the exception of yields of soybean roots which was nearly significant. The oat yields were not

TABLE 15

ANALYSES OF THE SOILS BEFORE ADDITION OF CALCIUM SULFATE AND LIME
1954

	Soil Type				
	Leon f.s.	Red Bay f.s.l.	Rutlege f.s.	Blanton f.s.	Gainesville l.f.s.
Sulfur extracted by Morgan's solution ppm. . . .	18.9	21.10	22.63	3.78	7.34
Water extractable sulfur ppm	13.03	1.80	9.47	0.77	0.06
Total organic and sulfate sulfur ppm.	100.0	152.6	233.0	42.0	75.6
Organic matter per cent	3.00	5.01	7.08	1.64	2.42
pH values	5.19	5.23	6.72	5.70	6.11
Soluble P_2O_5 lbs/acre	87	11	48	14	29
Exchangable CaO lbs/acre	1680	1960	4760	952	1960
Exchangable K_2O lbs/acre	148	366	134	72	96
Soluble NO_3 relative	Medium	High	High	Medium	Medium

TABLE 16

PLANT RESPONSES AND SOIL ANALYSES ON LEON FINE SAND TO WHICH SULFUR WAS ADDED AT FOUR RATES - AVERAGES OF TWO REPLICATIONS

	Rate of Sulfur Added lbs/acre				L.S.D.	
	0.00	1.86	7.44	29.76	.05	.01
PLANT RESPONSES						
Oats						
Catalase Activity[a]						
February 13, 1954	25.0	23.8	25.8	27.8	....	....
February 25, 1954	16.6	15.0	12.6	13.0	....	....
March 27, 1954	13.5	13.4	14.3	16.8	....	....
First Harvest						
Yields gms/jar	13.1	13.0	13.6	13.1	....	....
Percentages of sulfur	0.38	0.39	0.43	0.53	0.08	0.11
Second Harvest						
Yields gms/jar	11.8	12.2	10.8	12.4	....	....
Percentages of sulfur	0.26	0.26	0.28	0.35	0.09	....
MILLET						
Catalase Activity	25.4	27.2	13.0	23.0	....	....
Yields gms/jar	18.0	15.8	22.0	24.4	3.5	4.6
Percentages of sulfur	0.12	0.11	0.13	0.19	0.03	0.04
Roots						
Yields gms/jar	48.7	63.4	59.4	55.2	15.3	....
Percentages of sulfur	0.14	0.15	0.15	0.22	0.07	....
Hubam						
Catalase Activity	14.3	27.2	25.8	22.4	9.7	13.2
Yields gms/jar	11.6	10.4	13.3	12.0	2.2	2.9
Percentages of sulfur	0.30	0.28	0.30	0.40	0.09	0.12
Soybeans						
Catalase activity	20.7	15.1	31.5	35.7	....	....
Yields gms/jar	23.8	24.4	25.2	27.8	3.5	....
Percentages of sulfur	0.22	0.23	0.24	0.22	....	....
Roots						
Yields gms/jar	15.3	19.2	26.0	28.9	15.3	....
Percentages of sulfur	0.25	0.23	0.29	0.30	0.07	....
SOIL ANALYSES						
After Being Cropped with Oats and Millet						
Morgan's soluble sulfur ppm.	6.6	6.9	9.0	10.0	....	....
Water soluble sulfur ppm.	1.80	1.48	4.04	3.04	2.59	....
Total sulfur ppm.	71	76	76	76	....	....
Organic matter per cent	2.94	2.90	2.83	2.82	....	....
pH values	5.87	5.87	5.88	5.88	....	....
After Being Cropped with Hubam and Soybeans						
Morgan's soluble sulfur ppm.	7.3	6.2	6.6	21.5	6.3	8.6
Water soluble sulfur ppm.	3.78	2.40	4.88	14.90	2.59	3.50
Total sulfur ppm.	74	71	74	81	....	....
Organic matter per cent	2.77	2.65	2.72	2.83	....	....
pH values	5.62	5.58	5.70	5.57	....	....

[a] Arbitrary units of O_2 produced

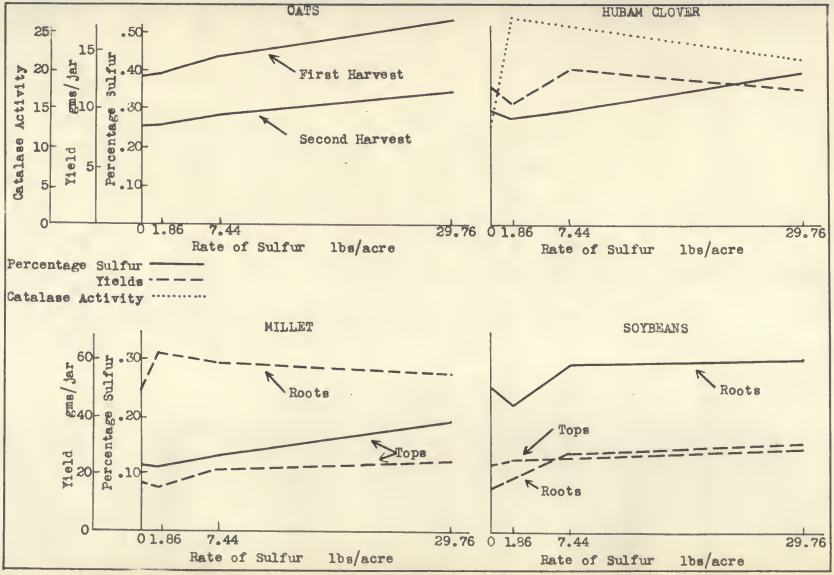

Fig. 4. Plant Responses on Leon Fine Sand to Which Four Rates of Sulfur Were Added

effected by the level of sulfur in Leon fine sand for either the first or second harvest (Photograph 14 and 15) but the sulfur contents of the plants were higher where sulfur had been added to the soil at the high rate. This indicates that luxury consumption was present in these plants and that none of the plants was deficient in sulfur. Apparently, catalase activity does not correlate with the amount of sulfur taken up in excess of that which is needed for normal growth of plants. The millet (Photograph 16) grown following the oats showed larger yields for the last two rates of sulfur additions than for the first two, but sulfur percentage of the plants was higher only where sulfur was added to the soil at the highest rate. Where sulfur had been added at the rate of 7.44 lbs. per acre there was a sufficient amount of sulfur in the soil for normal plant growth in the first part of the growing period but at the end of this period sulfur was deficient. Errors were large in the measurement of root yields but the trend was apparently an increase where a small amount of sulfur was added. The results from analyses showed no effect caused by sulfur treatments after being cropped with oats and millet but a significant difference was approached in the amounts of water soluble sulfur in the soil which were somewhat relative to the yields of millet with respect to sulfur applications. Catalase activity was lower in Hubam grown where no sulfur was added to the soil than where sulfur was added at the rate of 1.86 lbs. per acre and above. The

yields showed no logical trends following treatments but there was a highly significant increase in yields where sulfur was applied at the rate of 7.44 lbs. per acre as compared to 1.86 lbs. per acre. The sulfur content of the plants was lower where sulfur was added at the rate of 1.86 lbs. per acre compared to where sulfur additions were omitted but the higher rates caused a larger concentration of sulfur in the plants. A slightly paler green color was observed in the plants where no sulfur was added to the soil but was not a large enough contrast to show in Photograph 17. Soybeans (Photograph 18) gave increased yields of tops and roots and increased percentages of sulfur in the roots where sulfur had been applied to the soil at higher rates. There were no sulfur deficiency symptoms in any of the plants at the time of harvest which correlates with no differences in the percentages of sulfur in the tops and no difference in the catalase activity.

The absence of responses of oats to sulfur additions at four rates to Red Bay loamy fine sand and the high percentages of sulfur in the plants for both the first and second harvest indicates that there was no lack of sulfur for growth even where no sulfur was added to the soil (Table 17 and Photographs 19 and 20). Catalase activity measurements indicated similar conclusions. The millet (Photograph 21) grown after the oats had a higher content of sulfur in the plants where sulfur was added at the highest rate. There were no differences in yield and no sulfur deficiency symptoms

TABLE 17

PLANT RESPONSES AND SOIL ANALYSES ON RED BAY FINE SANDY LOAM TO WHICH SULFUR WAS ADDED AT FOUR RATES - AVERAGES OF TWO REPLICATIONS

	Rate of Sulfur Added lbs/acre				L.S.D.	
	0.00	1.86	7.44	29.76	.05	.01
PLANT RESPONSES						
Oats						
Catalase Activity[a]						
February 13, 1954	27.8	26.8	26.5	26.5	....	....
February 25, 1954	22.6	23.4	24.4	26.5	....	....
March 27, 1954	15.8	17.1	18.7	18.0	....	....
First Harvest						
Yields gms/jar	11.8	12.2	11.2	11.4	....	....
Percentages of sulfur	0.48	0.49	0.50	0.54	....	....
Second Harvest						
Yields gms/jar	13.4	14.2	14.4	15.4	....	....
Percentages of sulfur	0.41	0.46	0.39	0.45	....	....
Millet						
Catalase activity	23.1	38.7	23.3	46.6	....	....
Yields gms/jar	26.2	25.2	24.3	23.4	....	....
Percentages of sulfur	0.26	0.28	0.26	0.34	0.03	0.04
Roots						
Yields gms/jar	20.4	18.1	29.9	18.5	....	....
Percentages of sulfur	0.18	0.17	0.16	0.18	....	....
Hubam						
Catalase activity	17.3	13.8	28.0	30.9	9.7	13.2
Yields gms/jar	10.8	12.5	12.8	10.4	2.2	....
Percentages of sulfur	0.37	0.43	0.39	0.55	0.09	0.12
Soybeans						
Catalase activity	13.4	25.2	28.9	30.9	....	....
Yields gms/jar	26.2	23.8	24.0	26.3	....	....
Percentages of sulfur	0.26	0.29	0.26	0.28	0.03	....
Roots						
Yields gms/jar	11.9	13.4	13.2	16.3	....	....
Percentages of sulfur	0.21	0.22	0.20	0.22	....	....
SOIL ANALYSES						
After Being Cropped with Oats and Millet						
Morgan's soluble sulfur ppm.	19.8	23.4	17.6	20.5	....	....
Water soluble sulfur ppm.	1.35	1.65	1.08	1.25	....	....
Total sulfur ppm.	128	128	124	122	....	....
Organic matter per cent	4.56	4.66	4.51	4.60	....	....
pH values	5.50	5.50	5.46	5.44	....	....
After Being Cropped with Hubam and Soybeans						
Morgan's soluble sulfur ppm.	24.8	29.1	27.8	37.4	6.3	8.6
Water soluble sulfur ppm.	1.86	2.33	2.05	3.24	....	....
Total sulfur ppm.	134	143	139	139	....	....
Organic matter	4.55	4.47	4.56	4.58	....	....
pH values	5.41	5.36	5.36	5.36	....	....

[a] Arbitrary units of O_2 produced

appeared. This indicates that luxury consumption caused the difference in sulfur content in the plants. Hubam (Photograph 22) had a higher catalase activity in the plants where sulfur had been added at higher rates, but the sulfur content was higher only where sulfur was applied at the highest rate. Yield differences were small as was also true of the sulfur contents of the soybeans. No visible sulfur deficiencies were present in the soybeans (Photograph 23). Catalase activity measurements indicate that soybeans where no sulfur was added were growing less actively at harvest time. There were no deficiency symptoms in any of the crops and no differences in yield. There were higher sulfur contents of the plants where sulfur was added to the soil. These results indicate that sulfur was not deficient in this soil. There was an apparent reduction of total sulfur in the soil in quantities of about twenty to fifty pounds per acre where the soil was cropped with legumes and with grasses.

The abnormality of the Rutlege fine sand which was discussed on page 67 affected the results obtained in this experiment on Rutlege soil. The yields (Table 18), where erratic and just barely significant in the millet and soybean tops. Photograph 26 shows apparent yield differences in the green material which did not correspond to the air dry weights from these jars which were 18.1, 22.8, 16.9, and 22.0 gms. per jar with increasing rates of sulfur, respectively. The apparent differences between the appearance of the plants

TABLE 18

PLANT RESPONSES AND SOIL ANALYSES ON RUTLEGE FINE SAND TO WHICH SULFUR WAS ADDED AT FOUR RATES - AVERAGES OF TWO REPLICATIONS

	Rate of Sulfur Added lbs/acre				L.S.D.	
	0.00	1.86	7.44	29.76	.05	.01
PLANT RESPONSES						
Oats						
Catalase Activity[a]						
February 13, 1954	35.0	30.0	30.8	31.0	....	....
February 25, 1954	35.0	30.3	33.5	31.2	....	....
March 22, 1954	16.4	17.0	21.2	19.5	....	....
First Harvest						
Yields gms/jar	12.2	11.8	12.4	11.1	....	....
Percentages of sulfur	0.38	0.40	0.44	0.50	0.08	0.11
Second Harvest						
Yields gms/jar	9.0	7.8	9.4	8.4	....	....
Percentages of sulfur	0.35	0.30	0.40	0.42	0.09	0.12
Millet						
Catalase activity	77.1	80.1	72.3	79.6	....	....
Yields gms/jar	16.7	21.0	18.4	20.5	3.5	....
Percentages of sulfur	0.18	0.16	0.18	0.32	0.03	0.04
Roots						
Yields gms/jar	27.0	34.9	35.4	29.2	....	....
Percentages of sulfur	0.20	0.22	0.20	0.20	....	....
Hubam						
Catalase activity	36.0	34.4	30.3	28.4	....	....
Yields gms/jar	11.6	15.8	15.8	13.8	2.2	2.9
Percentages of sulfur	0.34	0.33	0.41	0.45	0.09	0.12
Soybeans						
Catalase activity	20.9	33.9	62.3	107.6	41.5	57.5
Yields gms/jar	16.4	14.0	12.8	15.8	3.5	....
Percentages of sulfur	0.28	0.30	0.30	0.31	0.03	....
Roots						
Yields gms/jar	8.6	10.2	15.8	12.8	....	....
Percentages of sulfur	0.28	0.24	0.28	0.40	0.07	0.10
SOIL ANALYSES						
After Being Cropped with Oats and Millet						
Morgan's soluble sulfur ppm.	20.6	21.1	25.5	21.2	....	....
Water soluble sulfur ppm.	1.96	1.85	1.26	4.27	2.59	....
Total sulfur ppm.	246	227	218	199	14	19
Organic matter per cent	6.70	6.72	7.38	7.10	....	....
pH values	6.82	6.86	6.88	6.86	....	....
After Being Cropped with Hubam and Soybeans						
Morgan's soluble sulfur ppm.	21.7	18.6	18.0	44.4	6.3	8.6
Water soluble sulfur ppm.	4.00	2.54	5.46	20.12	2.59	3.50
Total sulfur ppm.	180	238	236	230	14	19
Organic matter	6.70	7.32	6.88	6.83	0.34	0.48
pH values	6.52	6.46	6.52	6.62	....	....

[a] Arbitrary units of O_2 produced

and the air dry yields could have been caused by a difference in the moisture content of the green plants. The oats had a severe manganese deficiency (Photograph 24 and 25). The symptoms were the same as those listed on page 69. There was a highly significant increase in the sulfur content where sulfur had been applied to the soil at the highest rate. The Hubam (Photograph 27) showed no manganese deficiency and the yields and sulfur contents of the plants were both increased at the .01 level by the addition of sulfur to the soil. The soybeans were growing upright until they were moved for taking Photograph 28. Manganese was evidently the element which was deficient in the plants because at the time of harvest most of the deficiency symptoms had cleared up as a result of an application of manganese two weeks before harvest. Also, catalase activity in the soybeans at harvest time was increased with increasing rates of sulfur applications. Figure 5 presents, graphically, the plant responses on Rutlege soil. The soil analyses showed an apparent highly significant reduction in the amount of total sulfur after they were cropped with grasses where sulfur was applied at the highest rate. The reverse of this was true in the soils where legumes were grown; the reduction occurred where no sulfur was added. There were also larger amounts of extractable sulfate sulfur in the soil where sulfur was added at the rate of 29.76 lbs. per acre.

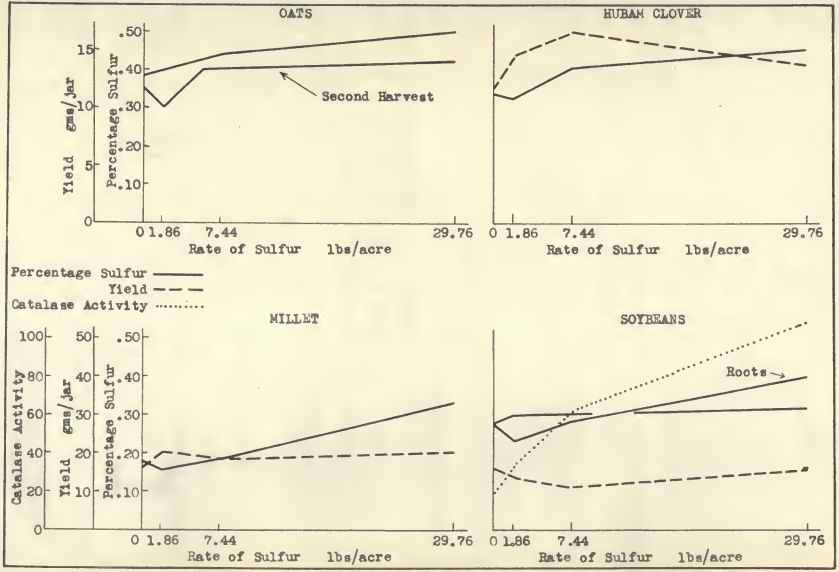

Fig. 5. Plant Responses on Rutlege Fine Sand to Which Four Rates of Sulfur Were Added

Plant responses to sulfur additions on Blanton fine sand (Table 19) were generally highly significant with respect to yields and sulfur content (Figure 6). Where no responses occurred, indications were that all plants were deficient in sulfur. Six weeks after planting oats and one week before harvests were made, sulfur deficiency symptoms were present in the oats where sulfur had been applied at the rates of 0 and 1.86 lbs. per acre. These are evident in Photograph 29. Catalase activity tended to correlate with the visual observations, but this determination was made before deficiency symptoms were visible. Before the second harvest of oats, severe sulfur deficiency symptoms were present where sulfur had been added at the rate of 0 and 1.86 lbs. per acre and a marked deficiency existed where 7.44 lbs. per acre had been applied (Photograph 30). Catalase activity agreed with these observations and with the yields. The effect of sulfur added to the soil on oats can be visualized from the response curves in Figure 6. Millet gave larger yields where sulfur had been added to the soil at the highest rate but there were no differences in the sulfur content at the time of harvest (Photograph 31). There was more root growth where more sulfur had been applied to the soil. The large yield of the millet for sulfur at the highest rate was made at the first part of the growing period because there was residual sulfur in the soil as can be seen from the sulfur contents of the previous crop of oats; but at harvest time all the plants were

TABLE 19

PLANT RESPONSES AND SOIL ANALYSES ON BLANTON FINE SAND TO WHICH SULFUR WAS ADDED AT FOUR RATES - AVERAGES OF TWO REPLICATIONS

	Rate of Sulfur Added lbs/acre				L.S.D.	
	0.00	1.86	7.44	29.76	.05	.01
PLANT RESPONSES						
Oats						
Catalase Activity[a]						
February 13, 1954	26.0	28.2	26.5	29.5	....	....
February 25, 1954	15.4	15.0	20.2	20.2	....	....
March 22, 1954	6.0	7.1	8.2	15.5	9.7	....
First Harvest						
Yields gms/jar	6.9	8.7	8.8	10.8	1.6	2.3
Percentages of sulfur	0.22	0.22	0.38	0.54	0.08	0.11
Second Harvest						
Yields gms/jar	6.0	7.5	8.5	10.2	2.2	2.9
Percentages of sulfur	0.22	0.21	0.24	0.39	0.09	0.12
Millet						
Catalase activity	14.2	11.3	27.3	37.3	....	....
Yields gms/jar	5.0	8.0	8.1	23.3	3.5	4.6
Percentages of sulfur	0.12	0.10	0.10	0.12	....	....
Roots						
Yields gms/jar	20.6	34.6	42.2	50.2	15.3	20.6
Percentages of sulfur	0.10	0.10	0.11	0.12	....	....
Hubam						
Catalase activity	6.9	13.6	20.0	14.4	9.7	13.2
Yields gms/jar	7.6	8.9	8.8	11.8	2.2	2.9
Percentages of sulfur	0.28	0.28	0.37	0.48	0.09	0.12
Soybeans						
Catalase activity	27.6	25.3	24.0	33.0	....	....
Yields gms/jar	9.2	10.2	14.6	16.6	3.5	4.6
Percentages of sulfur	0.16	0.14	0.20	0.25	0.03	0.04
Roots						
Yields gms/jar	10.9	16.6	11.2	18.6	....	....
Percentages of sulfur	0.16	0.12	0.17	0.30	0.07	0.10
SOIL ANALYSES						
After Being Cropped with Oats and Millet						
Morgan's soluble sulfur ppm.	4.2	4.0	4.8	4.0	....	....
Water soluble sulfur ppm.	2.20	1.44	2.13	2.86	....	....
Total sulfur ppm.	32	35	30	30	....	....
Organic matter per cent	1.60	1.64	1.57	1.52	....	....
pH values	5.33	5.96	5.95	6.01	....	....
After Being Cropped with Hubam and Soybeans						
Morgan's soluble sulfur ppm.	5.5	4.5	5.0	6.8	....	....
Water soluble sulfur ppm.	1.78	1.62	1.95	3.40	....	....
Total sulfur ppm.	37	38	36	40	....	....
Organic matter	1.62	1.57	1.58	1.54	....	....
pH values	5.96	5.99	5.98	5.96	....	....

[a] Arbitrary units of O_2 produced

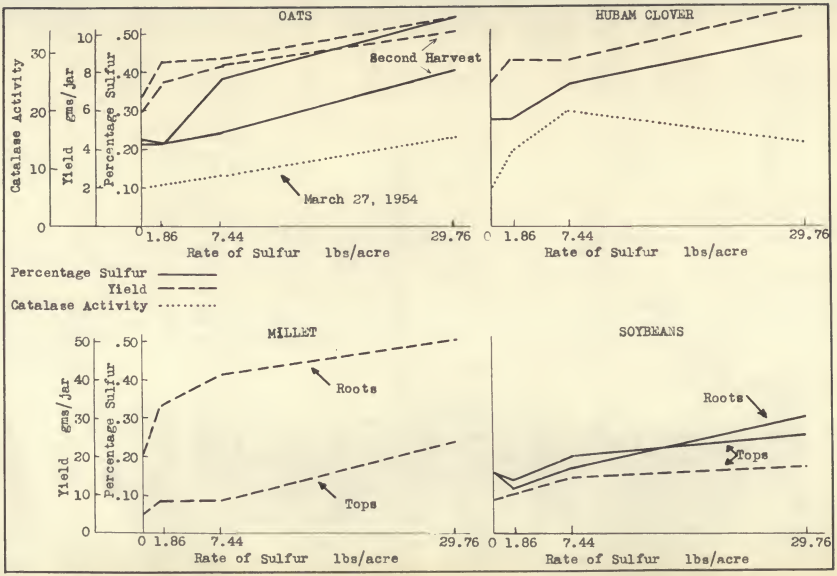

Fig. 6. Plant Responses of Blanton Fine Sand to Which Four Rates of Sulfur Were Added

insufficently supplied with sulfur as the soil and plant analyses show.

Yields and sulfur contents of Hubam increased where sulfur was applied at the higher rate to the Blanton fine sand (Table 19). Extreme sulfur deficiency symptoms existed in the plants where no sulfur was added but none was present where sulfur had been added at the highest rate (Photograph 32). The plants growing where sulfur had been added at the other rates showed definite deficiencies of sulfur. After the soil had been cropped with Hubam, there was sufficient quantity of residual sulfur in the soil where sulfur had been added at the two highest rates to increase the yields and sulfur content of soybeans. Ten days before time of harvest they showed a definite color graduation with sulfur additions (Photograph 33). At the time of harvest, the trends of catalase activity in the plant indicated that there was a difference in the growth rate between the two highest sulfur additions.

Plant responses on the Gainesville loamy fine sand (Table 20 and Figure 7) to which sulfur was added at four rates were greater in the first crops than in the second, especially, with respect to sulfur content of the plants. The trends in catalase activity show that the plants became deficient in sulfur after five and before six and one-half weeks after planting where sulfur was applied at the three lowest rates. One week before the first harvest of oats

TABLE 20

PLANT RESPONSES AND SOIL ANALYSES ON GAINESVILLE LOAMY FINE SAND TO WHICH SULFUR WAS ADDED AT FOUR RATES - AVERAGES OF TWO REPLICATIONS

	Rate of Sulfur Added lbs/acre				L.S.D.	
	0.00	1.86	7.44	29.76	.05	.01
PLANT RESPONSES						
Oats						
Catalase Activity[a]						
February 13, 1954	26.2	22.8	25.2	22.5	....	....
February 25, 1954	13.2	13.6	18.9	31.2	....	....
March 27, 1954	9.4	7.0	10.6	18.4	9.7	....
First Harvest						
Yields gms/jar	7.8	10.2	11.5	11.4	1.6	2.3
Percentages of sulfur	0.18	0.21	0.29	0.58	0.08	0.11
Second Harvest						
Yields gms/jar	7.6	6.0	8.8	8.9	2.2	2.9
Percentages of sulfur	0.28	0.24	0.28	0.44	0.09	0.12
Millet						
Catalase activity	27.7	17.0	18.2	35.5	....	....
Yields gms/jar	9.4	7.0	9.4	25.2	3.5	4.6
Percentages of sulfur	0.10	0.10	0.10	0.10	....	....
Roots						
Yields gms/jar	25.3	12.4	30.2	38.6	15.3	20.6
Percentages of sulfur	0.13	0.12	0.17	0.12	....	....
Hubam						
Catalase activity	10.2	19.2	16.6	24.0	9.7	13.2
Yields gms/jar	13.3	15.2	19.2	17.3	2.2	2.9
Percentages of sulfur	0.27	0.42	0.33	0.46	0.09	0.12
Soybeans						
Catalase activity	27.1	31.4	28.7	51.5	....	....
Yields gms/jar	12.4	11.5	15.0	25.2	3.5	4.6
Percentages of sulfur	0.13	0.14	0.13	0.20	0.03	0.04
Roots						
Yields gms/jar	34.3	16.6	31.5	28.3	15.3	....
Percentages of sulfur	0.12	0.15	0.14	0.16	....	....
SOIL ANALYSES						
After Being Cropped with Oats and Millet						
Morgan's soluble sulfur ppm.	5.6	6.1	6.2	6.6	....	....
Water soluble sulfur ppm.	1.33	1.00	1.25	1.26	....	....
Total sulfur ppm.	70	68	69	73	....	....
Organic matter per cent	2.04	2.02	2.06	2.08	....	....
pH values	6.26	6.11	6.15	6.15	....	....
After Being Cropped with Hubam and Soybeans						
Morgan's soluble sulfur ppm.	5.2	4.4	5.6	5.2	....	....
Water soluble sulfur ppm.	1.43	1.00	1.43	0.92	....	....
Total sulfur ppm.	69	67	68	69	....	....
Organic matter per cent	2.04	1.96	2.06	2.05	....	....
pH values	6.14	6.12	6.23	6.18	....	....

[a] Arbitrary units of O_2 produced

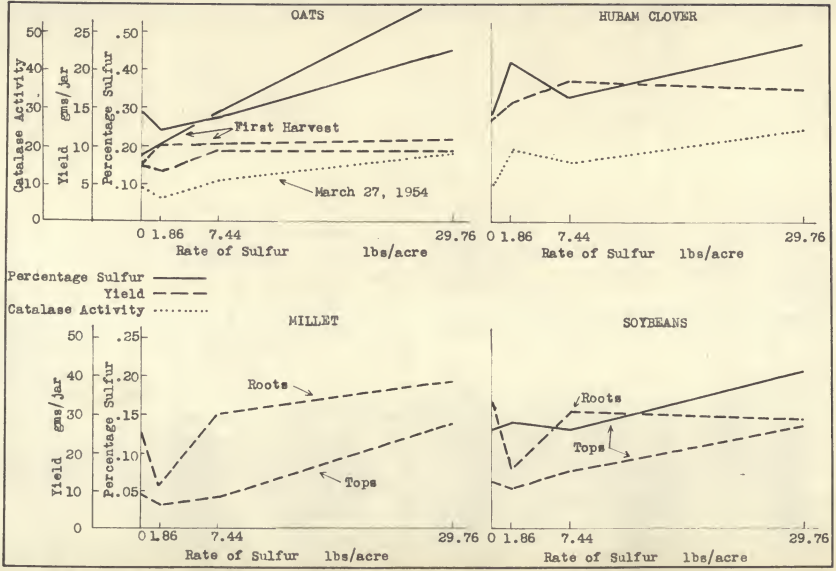

Fig. 7. Plant Responses on Gainesville Loamy Fine Sand to Which Four Rates of Sulfur Were Added

(Photograph 34) sulfur deficiency symptoms appeared where sulfur had been applied at the rates of 0 and 1.86 lbs. per acre. The yields were lower where sulfur was not applied, but there was an increase in sulfur content in the plants where sulfur was added at increasing rates. Photograph 35 shows that visual plant responses were indicative of the yields of dry material and sulfur contents. Catalase activity, yield and sulfur contents were lower where a small amount of sulfur was added but the latter was the only one significantly so. With larger additions of sulfur, these responses were increased. The same trends existed in the millet responses (Photograph 36), both tops and roots, except there was no significant differences in sulfur contents. The plants where the highest rate of sulfur was applied showed deficiency symptoms when they were harvested. Hubam (Photograph 37) generally responded positively to increased levels of sulfur in the soil. The sulfur content of the Hubam grown where sulfur was added to the soil at the rate of 1.86 lbs. per acre may be in error since the plants from replication two contained, as an average of triplicate analyses, 0.56 per cent sulfur while the plants in replication one contained 0.28 per cent sulfur. The soybeans (Photograph 38) grown after Hubam showed sulfur deficiency symptoms in all the plants only ten days after planting except where the highest rate of sulfur had been added. Catalase activity was in agreement with these observations and with the sulfur content of these plants.

The same trends existed in the responses of soybeans, as can be seen in the photograph, as were present with millet; that is, negative yield response existed where small quantities of sulfur had been added. However, there was enough residual sulfur in the soil, which had received the sulfur addition at the highest rate, to prevent a sulfur deficiency in the plants. This is varified by catalase activity measurements, yields, and sulfur content of the soybeans.

The uptake of sulfur by the plants where sulfur had been added was larger than where no sulfur was added. The amounts of sulfur taken-up by the plants (Table 21) grown on each of the soils were greater than the amounts added to the soils. The highest rate of sulfur application caused over three and one-half times as much uptake as where none was added. The sulfur which was taken-up for the higher rates came from that which was applied because the amount of sulfur comming from the soil after allowing for the added sulfur was usually about equal to or lower than that where no sulfur was added. The exception to this is the Blanton soil where grasses were grown in which case larger sulfur additions caused larger amounts of sulfur to be taken from the soil.

From the "b" values in Tables 30, 31 and 32 it was calculated that the addition of 30 lbs. of sulfur per acre to Leon, Blanton, and Gainesville soils caused an increase of 0.10 per cent sulfur in the plants and an increase in yields of 990 lbs. per acre on the average.

TABLE 21

UPTAKE OF SULFUR BY GRASSES AND LEGUMES GROWN ON FIVE SOIL TYPES TO WHICH SULFUR WAS ADDED AT FOUR RATES - AVERAGES OF TWO REPLICATIONS

Soil Type	Type of Crop	Amounts of Sulfur Added gms/jar			
		0.000	0.007	0.028	0.111
		Total Uptake of Sulfur gms/jar			
Leon f.s.	Grasses	0.172	0.195	0.202	0.284
	Legumes	.124	.128	.173	.195
Red Bay f.s.l.	Grasses	.216	.226	.225	.243
	Legumes	.132	.153	.139	.165
Rutlege f.s.	Grasses	.162	.184	.197	.212
	Legumes	.110	.120	.148	.163
Blanton f.s.	Grasses	.052	.076	.109	.188
	Legumes	.052	.066	.080	.146
Gainesville l.f.s.	Grasses	.078	.059	.121	.178
	Legumes	0.093	0.101	0.121	0.176
		Total Uptake Minus Amount Added gms/jar			
Leon f.s.	Grasses	0.172	0.188	0.174	0.173
	Legumes	.124	.121	.145	.084
Red Bay f.s.l.	Grasses	.216	.219	.197	.132
	Legumes	.132	.146	.111	.054
Rutlege f.s.	Grasses	.162	.177	.169	.101
	Legumes	.110	.113	.120	.052
Blanton f.s.	Grasses	.052	.069	.081	.077
	Legumes	.052	.055	.052	.035
Gainesville l.f.s.	Grasses	.078	.052	.093	.067
	Legumes	0.093	0.094	0.093	0.065

Correlation of Plant Responses and Chemical Analyses for Sulfur of Soil Profiles - 1955

The response of plants to native sulfate sulfur in profile sections of the Blanton fine sand (Table 22) correlates well with the amounts of sulfur in the soil profile as determined by chemical methods. The oats at harvest time (Photograph 39) showed sulfur deficiency symptoms in all the plants except the ones growing on the section of the profile which was 30-42 inches deep. The deficiencies were further established by the yields which were about eighty-five per cent higher and by the percentage of sulfur in the plants which was over three times that of the plants for the upper portions of the profile. The crop of soybeans which followed the oats showed a different response in that the yields of the plants on the surface layer were higher. The sulfur deficiency symptoms were quite severe in the plants grown on soil from the 6-18 and 18-30 inch depths (Photograph 40). The soybeans grown on the 30-42 inch section contained much higher amounts of sulfur.

Total uptake of sulfur by the plants as grams per jar was almost identical to the amounts of Morgan's solution extractable sulfur when expressed in grams per jar (Table 22), calculated as 8,000 gms. of air dry soil per jar. A comparison of the amounts of extractable sulfate sulfur with the amounts of total sulfur in the soil shows that about one-eighth of the total sulfur was available except in the

TABLE 22

PLANT RESPONSES AND SOIL ANALYSES ON BLANTON FINE SAND PROFILE

	Depth of Soil inches			
	0-6	6-18	18-30	30-42
Plant Responses[a]				
Oats				
Yields gms/jar[b]	5.3	5.3	4.9	9.2
Percentages of sulfur[c]	0.074	0.076	0.075	0.259
Soybeans				
Yields gms/jar[d]	11.4	6.2	7.3	8.1
Percentages of sulfur[e]	0.058	0.044	0.044	0.125
Total uptake of sulfur gms/jar	0.032	0.020	0.021	0.102
Soil Analyses				
Water extractable sulfur ppm.	3.61	2.08	0.07	2.22
Morgan's soluble sulfur ppm.	4.89	3.33	2.66	13.11
Morgan's soluble sulfur gms/jar	0.035	0.027	0.021	0.105
Total organic and sulfate sulfur ppm.	30.4	20.0	18.4	44.0
Total organic and sulfate sulfur gms/jar	0.243	0.160	0.147	0.352
Mechanical analyses				
Sands	94.0	93.5	94.0	88.0
Coarse silt	2.4	1.8	1.4	0.6
Fine silt	0.7	1.3	1.2	2.6
Clay	2.9	3.4	3.4	8.8

[a] Averages of three replications
[b] L.S.D. .05, 1.2 ; .01, 1.8 C.V., 9.2 per cent
[c] L.S.D. .05, 0.034 ; .01, 0.040 C.V., 12.2 per cent
[d] L.S.D. .05, 3.26 ; .01, 4.50 C.V., 21.4 per cent
[e] L.S.D. .05, 0.006 ; .01, 0.008 C.V., 8.8 per cent

30-42 inch depth where about one-third of the total sulfur was available.

The soil sulfur profile characteristics of Gainesville loamy fine sand (Table 23) are widely different from those of the Blanton fine sand. Some of the differences are caused by the presence of a sharp break into a clay pan at about forty-two inches in the Blanton soil and a gradual increase in the clay content with depth in the Gainesville profile. The oats on the surface six inches showed sulfur deficiency symptoms at the time of harvest (Photograph 41). The yield differences are apparent but not large; however, the sulfur contents of these plants differed greatly in that the lowest sulfur content occurred in the plants grown on the surface six inches. For the 6-18 to the 30-42 inch depths there were no differences in the sulfur content of the plants but there was less sulfur in the plants grown on the soil of the 42-54 inch depth. The yields of soybeans were lower where they were grown on soil from the deeper portions of the profile. Sulfur deficiency symptoms were present in the soybeans grown on the surface soil and on the 42-54 inch section of the profile (Photograph 42). Figure 8 shows graphically the yields and percentages of sulfur in the oats and soybeans, (A); and a graph, (B), shows the amounts of extractable sulfur in the soil compared to the amounts that were taken up by the plants. The amount of extractable sulfur was larger or equal to the amount taken-up by the plants. The amount of extractable

TABLE 23

PLANT RESPONSES AND SOIL ANALYSES ON GAINESVILLE LOAMY FINE SAND PROFILE

	Depth of Soil inches				
	0-6	6-18	18-30	30-42	42-54
Plant Responses[a]					
Oats					
Yields gms/jar[b]	10.9	8.2	9.1	9.4	10.0
Percentages of sulfur[c]	0.095	0.204	0.216	0.190	0.166
Soybeans					
Yields gms/jar[d]	11.2	10.6	9.5	8.8	5.8
Percentages of sulfur[e]	0.064	0.105	0.084	0.066	0.051
Total uptake of sulfur gms/jar	0.053	0.084	0.082	0.071	0.059
Soil Analyses					
Water soluble sulfur ppm.	3.55	2.68	1.80	1.88	1.80
Morgan's soluble sulfur ppm.	5.56	15.11	12.90	10.00	7.35
Morgan's soluble sulfur gms/jar	0.044	0.121	0.113	0.080	0.059
Total organic and sulfate sulfur ppm.	67.6	46.6	36.0	38.8	38.8
Total organic and sulfate sulfur gms/jar	0.541	0.373	0.288	0.310	0.310
Mechanical Analyses					
Sands	86.4	86.9	84.0	77.7	77.7
Coarse silt	6.3	6.8	3.9	6.3	6.4
Fine silt	0.9	0.9	0.7	0.2	0.2
Clay	6.4	7.0	11.4	15.8	15.7

[a] Averages of three replications
[b] L.S.D. .05, 1.2 ; .01, 1.8 C.V., 9.2 per cent
[c] L.S.D. .05, 0.034 ; .01, 0.040 C.V., 12.2 per cent
[d] L.S.D. .05, 3.3 ; .01, 4.5 C.V., 21.4 per cent
[e] L.S.D. .05, 0.006 ; .01, 0.008 C.V., 8.8 per cent

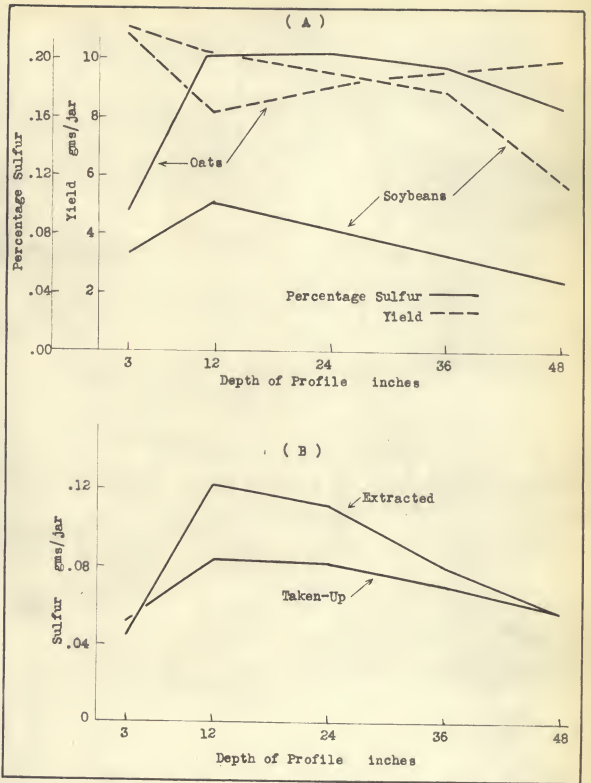

Fig. 8. (A) Plant Responses on Sections of the Gainesville Loamy Fine Sand Profile and (B) the Relation of the Amount of Extractable Sulfur to the Amount Taken-up by Plants.

sulfur was larger or equal to the amount taken up by the plants except in the surface where some of the organic form was probably converted into available form during the growing period of the plants. The amounts of total sulfur decreased with an increase in depth of the profile.

SUMMARY AND CONCLUSIONS

A method was modified which made it possible to determine the amount of sulfate sulfur in soils that is extractable by a buffered acetic acid solution. The modified method gives relative values as to the amount of available sulfate sulfur in soils.

Water extractable sulfate does not indicate the amount of available sulfur, especially in soils with a heavy texture. The ratio of organic matter to total sulfur in soil was about 330. In all the soils tested, about 10 per cent of the total sulfur was extractable except in Leon fine sand where 20 per cent was obtained. Sulfur uptake by plants correlated with extractable sulfur. Sulfur characteristics of the soil profile varied with soil types. Total and extractable sulfur decreased with an increase in depth to thirty inches in a Blanton fine sand. Below this depth the largest amounts of sulfur were present. In a Gainesville loamy fine sand, extractable sulfate sulfur was lowest, 5.56 ppm., in the surface six inches. Thereafter, sulfur decreased from 15.11 ppm. in the 6-18 inch depth to 7.35 ppm. in the 42-50 inch depth. Total organic and sulfate sulfur decreased with an increase in depth to 30 inches, but was

uniform from this depth to 54 inches.

Catalase activity was useful in determining the relative rates of growth or metabolic processes in the plants. The measurements materially supplemented other criteria.

Leon, Blanton, and Gainesville soils were deficient in sulfur as indicated by increased yields and sulfur contents of grasses and legumes where sulfur was added to the soil. Calculations showed that on an average the addition of sulfur to these soils at the rate of 30 lbs. per acre increased the sulfur content of the plants 0.10 of a per cent and increased the yields 990 lbs. per acre. The addition of sulfur to Red Bay soil did not generally cause increased crop yields, but it did cause higher percentages of sulfur to be present in the plants. The critical levels of sulfur in Hubam sweetclover, oats, hairy indigo, soybeans, millet, and pangolagrass were found to be about 0.30, 0.25, 0.20, 0.20, 0.15, and 0.13 per cent, respectively.

Representative soils of North-Central Florida have been shown, by greenhouse experiments, to contain insufficient quantities of sulfur to support normal plant growth. There are several factors which lead to the opinion that the deficiencies would be as severe, or more severe, under field conditions. Leaching losses of sulfur would be large. The amount of sulfur brought down by rainwater is small in places, as little as three pounds per acre per year. The transformations of organic sulfur into available sulfur would be

more rapid in the greenhouse, because of soil disturbance, higher temperatures and more favorable moisture conditions. However, for crops under irrigation, sufficient sulfur may be added in the irrigation water in some instances.

Further study should be made of methods for determining presence of available sulfur. Investigation is needed with reference to various crops, especially vegetables, relative to methods of application, time and frequency of application, amount, and source of sulfur which should be applied.

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APPENDIX

TABLE 24

LOG OF OPERATIONS ON LEON FINE SAND AND RED BAY FINE SANDY LOAM
1953

Date	Operation
January 15, 1953	Applied nutrient solution:nitrogen phosphorus and potassium.
January 16, 1953	Applied gypsum.
January 19, 1953	Planted oats and Hubam sweet clover.
February 6, 1953	Planted pangolagrass.
February 19, 1953	Applied 1 gm. KNO_3 per jar on oats and pangolagrass.
February 26, 1953	Determined catalase activity in oats.
March 5, 1953	Determined catalase activity in oats and pangolagrass and applied 1 gm. K_2HPO_4 per jar.
March 12, 1953	Harvested oats and pangolagrass.
March 28, 1953	Applied 1 gm. NH_4NO_3, KNO_3, and KH_2PO_4 per jar except on Hubam.
April 4, 1953	Took photographs, black and white and 2" X 2" color slides; and harvested Hubam.
April 8, 1953	Harvested pangolagrass.
April 17, 1953	Harvested Hubam.
April 20, 1953	Planted hairy indigo.
May 7, 1953	Harvested pangolagrass.
May 9, 1953	Photographed oats, 2" X 2" color slides.
May 22, 1953	Harvested oats.
May 25, 1953	Applied 1 gm. KNO_3 per jar on hairy indigo and pangolagrass.

(continued)

TABLE 24 (continued)

LOG OF OPERATIONS ON LEON FINE SAND AND RED BAY FINE SANDY LOAM
1953

Date	Operation
June 2, 1953	Harvested pangolagrass.
June 4, 1953	Sampled soil in oats jars.
June 6, 1953	Applied 1 gm. NH_4NO_3 per jar on hairy indigo and pangolagrass.
June 15, 1953	Determined catalase activity in pangolagrass and hairy indigo.
June 17, 1953	Took photographs, black and white and color.
June 18, 1953	Harvested pangolagrass and hairy indigo.
June 29, 1953	Sampled soil in hairy indigo and pangolagrass jars.

TABLE 25

LOG OF OPERATIONS ON RUTLEGE FINE SAND, BLANTON FINE SAND, AND GAINESVILLE LOAMY FINE SAND - 1953

Date	Operation
February 18, 1953	Applied lime and nutrient elements except gypsum.
February 19, 1953	Planted oats, Hubam sweet clover, and pangolagrass.
February 21, 1953	Applied gypsum.
March 28, 1953	Applied 1 gm. NH_4NO_3, KNO_3, PH_2PO_4 per jar except on Hubam.
April 4, 1953	Took photographs black and white and 2"X2" color slides; and harvested pangolagrass.
April 13, 1953	Applied 1 gm. MgO per jar on Rutlege soil.
April 17, 1953	Harvested pangolagrass.
April 20, 1953	Harvested Hubam and planted hairy indigo.
May 7, 1953	Harvested pangolagrass.
May 9, 1953	Took photographs, 2" X 2" color slides.
May 22, 1953	Harvested oats.
May 25, 1953	Applied 1 gm. KNO_3 per jar on hairy indigo and pangolagrass.
June 2, 1953	Harvested pangolagrass.
June 4, 1953	Sampled soil in oats jars.
June 6, 1953	Applied 1 gm. NH_4NO_3 per jar on hairy indigo and pangolagrass.
June 15, 1953	Determined catalase activity in hairy indigo and pangolagrass.
June 17, 1953	Took photographs, black and white and color.
June 18, 1953	Harvested pangolagrass and hairy indigo.
June 29, 1953	Sampled soil in hairy indigo and pangolagrass jars.

TABLE 26

LOG OF OPERATIONS OF THE SULFUR RATE EXPERIMENT - 1954

Date	Operation
December 22, 1953	Limed Leon and Red Bay soils, 1,000 lbs/acre.
January 1, 1954	Applied nutrients equivalent to 2,000 lbs/acre of a 6-6-6 fertilizer and 50 lbs/acre of $MgCl_2 \cdot 6H_2O$ and gypsum in solution.
January 10, 1954	Planted oats and Hubam sweet clover.
February 13, 1954	Determined catalase activity in oats.
February 25, 1954	Determined catalase actiivity in oats.
March 1, 1954	Photographed oats and Hubam, black and white and 2"X2" color slides; and harvested oats.
March 15, 1954	Applied 1.25 gms. of KNO_3 per jar where oats was growing.
March 27, 1954	Determined catalase activity in oats and Hubam.
April 1, 1954	Photographed, black and white and 2"X2" color slides and harvested all jars.
April 13, 1954	Mixed the soil in each jar.
April 14, 1954	Planted soybeans and millet.
April 19, 1954	Replanted soybeans.
April 24, 1954	Applied 1 gm. of KNO_3 to each jar.
May 5, 1954	Sprayed soybeans and millet growing on Rutlege soil with colloidel magnanese suspension.
May 8, 1954	Applied nutrient solution equivalent to 1,000 lbs/acre of a 6-6-6 fertilizer.
May 20, 1954	Took photographs, black and white and color slides.
May 21, 1954	Determined catalase activity and harvested soybeans and millet.
June 18, 1954	Harvested roots and sampled soils.

TABLE 27

LOG OF OPERATIONS OF THE GREENHOUSE PROFILE EXPERIMENT
1955

Date	Operation
February 1, 1955	Limed and moistened the soils.
February 3, 1955	Applied nitrogen, phosphorus, potassium, and minor elements.
February 17, 1955	Planted oats.
March 23, 1955	Applied 1 gm. NH_4NO_3 per jar.
April 5, 1955	Took photographs, black and white.
April 10, 1955	Harvested oats.
April 26, 1955	Cultivated.
May 5, 1955	Planted soybeans.
June 16, 1955	Took photographs, black and white.
June 19, 1955	Harvested soybeans.

TABLE 28

COEFFICIENTS OF VARIATION FROM THE STATISTICAL ANALYSES OF THE PLANT RESPONSES AND SOIL ANALYSES FOR THE 1954 GREENHOUSE EXPERIMENT

	C.V., $(\frac{s}{\bar{x}})100$	
	Error b	Error c[a]
Plant responses		
Catalase activity in oats February 13, 1954	12.1	....
Catalase activity in oats February 25, 1954	20.1	....
Yields of oats, first harvest	6.8	....
Total sulfur in oats, first harvest	9.1	....
Catalase activity in oats and Hubam	19.1	26.8
Yields of Hubam and second harvest of oats	23.0	9.2
Total sulfur in Hubam and second harvest of oats	30.3	12.6
Catalase activity in soybeans and millet	29.0	....
Total sulfur in soybeans and millet	10.2	7.4
Yields of soybeans and millet	13.8	9.4
Yields of roots of soybeans and millet	29.1	28.4
Total sulfur of roots of soybeans and millet	19.7	19.2
Soil Analyses		
Sulfur extracted by Morgan's solution	21.5	22.6
Sulfur extracted by water	30.0	42.3
Total organic and sulfate sulfur	37.6	6.5
Organic matter content	3.3	4.8
pH values	1.3	1.1

[a] Sources of variation and degrees of freedom (d.f.)

TYPICAL

Source	d.f.
Total	79
Main plots	3
Replications	1
Crops (C)	1
Error a	1
Sub plots in main plots	16
Soil types (S)	4
S X C	4
Error b	8
Sub-sub plots in sub plots	60
Rates of sulfur (R)	3
R X S	12
R X C	3
R X S X C	12
Error c	30

ONE CROP

Source	
Total	39
Main plots	9
Replications	1
Soil types (S).	4
Error a	4
Sub plots	30
Rates of sulfur (R)	3
R X S	12
Error b	15

TABLE 29

ANALYSIS OF VARIANCE USED IN REGRESSION ANALYSES FOR EXAMINING THE POLYNOMIAL TRENDS OF THE DEPENDENT VARIABLES ON SULFUR RATES ON LEON FINE SAND, BLANTON FINE SAND, AND GAINESVILLE LOAMY FINE SAND (TABLES 30, 31, and 32)

Source	Degrees of Freedom	Mean Square
Between rates of sulfur	3	M.S. (rates)
Linear regression on sulfur	1	M.S. (reg.)
Deviation from linearity	2	M.S. (res.)
Error	15 or 30	s_e^2

TABLE 30

POLYNOMIAL TRENDS OF THE DEPENDENT VARIABLES, PLANT RESPONSES, ON THE RATE OF SULFUR INCLUDING REGRESSION COEFFICIENTS ON LEON FINE SAND

	M.S.(rates)	M.S.(reg.)	M.S.(res.)	s_e^2	b^a
Oats					
Percentage sulfur, first harvest	0.0094	0.0280**	0.0001	0.0013	0.0050
Percentage sulfur, second harvest	0.0037	0.0109*	0.0000	0.0019	.0031
Millet					
Yields gms/jar	30.00**	65.58**	12.08	2.95	.2420
Percentage sulfur	0.0026**	0.0075**	0.0002	0.0002	.0026
Hubam					
Yields gms/jar	2.87	0.65	3.66	1.10	.0240
Percentage sulfur	0.0059	0.0164**	0.0006	0.0019	.0038
Soybeans					
Yields					
Tops gms/jar	6.51	18.40*	0.57	2.95	.1278
Roots gms/jar	65.29	184.4	5.88	56.03	.4047
Percentage sulfur, roots	0.0069*	0.0081*	0.0063*	0.0013	-0.0027

[a] Regression coefficients as units of plant responses per pound of sulfur added per acre. To convert grams per jar to pounds per acre, multiply by 267.

TABLE 31

POLYNOMIAL TRENDS OF THE DEPENDENT VARIABLES, PLANT RESPONSES, ON THE RATE OF SULFUR INCLUDING REGRESSION COEFFICIENTS ON BLANTON FINE SAND

	M.S.(rates)	M.S.(reg.)	M.S.(res.)	s^2_e	b^a
Oats					
Catalase activity March 27, 1954	9.22	27.54	0.04	23.11	0.1564
First Harvest					
Yields gms/jar	1.27	3.13*	0.23	0.589	.0527
Percentage sulfur	0.0117**	0.0322**	0.0015	0.0013	.0054
Second Harvest					
Yields gms/jar	1.56	3.90	0.38	1.10	.0589
Percentage sulfur	0.0036	0.0105	0.0001	0.0019	.0030
Millet					
Yields					
Tops gms/jar	34.11**	99.65**	1.34	2.95	.2975
Roots gms/jar	82.66	164.93	36.52	56.03	.3827
Hubam					
Yields	1.59	4.46	0.16	1.10	.0630
Percentage sulfur	0.0045	0.0127*	0.0004	0.0019	.0034
Soybeans					
Yields tops gms/jar	6.22	14.44*	2.11	2.95	.1132
Percentage Sulfur					
Tops	0.0012*	0.0031**	0.0002	0.0002	.0017
Roots	0.0030	0.0085*	0.0003	0.0013	0.0027

[a] Regression coefficients as units of plant responses per pound of sulfur added per acre. To convert grams per jar to pounds per acre, multiply by 267.

TABLE 32

POLYNOMIAL TRENDS OF THE DEPENDENT VARIABLES, PLANT RESPONSES, ON THE RATE OF SULFUR INCLUDING REGRESSION COEFFICIENTS ON GAINESVILLE LOAMY FINE SAND

	M.S.(rates)	M.S.(reg.)	M.S.(res.)	s_e^2	b^a
Oats					
Catalase activity March 27, 1954	12.16	34.17	1.16	23.11	0.1742
First Harvest					
Yields gms/jar	1.481	1.750	1.474	0.589	.0394
Percentage sulfur	0.0167**	0.0501**	0.0000	0.0013	.0067
Second Harvest					
Yields gms/jar	0.92	1.14	0.80	1.10	.0318
Percentage of sulfur	0.0029	0.0109*	0.0004	0.0019	.0031
Millet					
Yields					
Tops gms/jar	35.08**	99.53**	2.86	2.95	.2973
Roots gms/jar	60.05	112.87	33.63	56.03	.3166
Hubam					
Catalase activity	16.54	32.02	8.80	23.11	.1686
Yields gms/jar	3.26	2.33	3.73	1.10	.0455
Percentage sulfur	0.0037	0.0054	0.0028	0.0019	.0022
Soybeans					
Tops					
Yields gms/jar	19.81*	58.65**	0.38	2.95	.2282
Percentage sulfur	0.0006	0.0016**	0.0001	0.0002	.0012
Root yields gms/jar	30.26	6.22	84.57	56.03	0.0235

[a] Regression coefficients as units of plant responses per pound of sulfur added per acre. To convert grams per jar to pounds per acre, multiply by 267.

Photograph 1. Oats Growing on Leon Fine Sand to Which Sulfur Was Added at the Rates of 0 and 4.65 lbs/acre, Left to Right. April 4, 1953.

Photograph 2. Pangolagrass Growing on Leon Fine Sand to Which Sulfur Was Added at the Rates of 0 and 4.65 lbs/acre, Left to Right. April 4, 1953.

Photograph 3. Pangolagrass Growing on Leon Fine Sand to Which Sulfur Was Added at the Rates of 4.65 and 0 lbs/acre, Left to Right. June 17, 1953.

Photograph 4. Oats Growing on Red Bay Fine Sandy Loam to Which Sulfur Was Added at the Rates of 0 and 4.65 lbs/acre, Left to Right. April 4, 1953.

Photograph 5. Pangolagrass Growing on Red Bay Fine Sandy Loam to Which Sulfur Was Added at the Rates of 4.65 and 0 lbs/acre, Left to Right. June 17, 1953.

Photograph 6. Oats Growing on Blanton Fine Sand to Which Sulfur Was Added at the Rates of 0 and 4.65 lbs/acre, Left to Right. April 4, 1953.

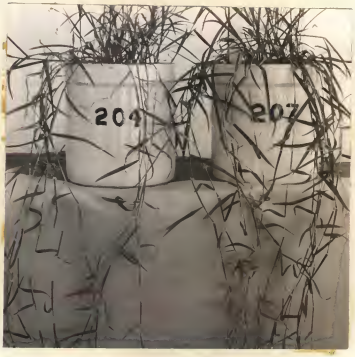
Photograph 7. Hairy Indigo Growing on Blanton Fine Sand to Which Sulfur Was Added at the Rates of 4.65 and 0 lbs/acre, Left to Right. June 17, 1953.

Photograph 8. Pangolagrass Growing on Blanton Fine Sand to Which Sulfur Was Added at the Rates of 0 and 4.65 lbs/acre, Left to Right. April 4, 1953.

Photograph 9. Oats Growing on Gainesville Loamy Fine Sand to Which Sulfur Was Added at the Rates of 0 and 4.65 lbs/acre, Left to Right. April 4, 1953.

Photograph 10. Hubam Growing on Gainesville Loamy Fine Sand to Which Sulfur Was Added at the Rates of 0 and 4.65 lbs/acre, Left to Right. April 4, 1953.

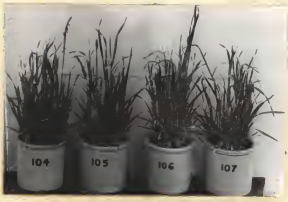
Photograph 11. Hairy Indigo Growing on Gainesville Loamy Fine Sand to Which Sulfur Was Added at the Rates of 4.65 and 0 lbs/acre, Left to Right. June 17, 1953.



Photograph 12. Pangolagrass Growing on Gainesville Loamy Fine Sand to Which Sulfur Was Added at the Rates of 0 and 4.65 lbs/acre, Left to Right. April 4, 1953.

Photograph 13. Pangolagrass Growing on Gainesville Loamy Fine Sand to Which Sulfur Was Added at the Rates of 4.65 and 0 lbs/acre, Left to Right. June 17, 1953.

Photograph 14. Oats Growing on Leon Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. March 1, 1954.



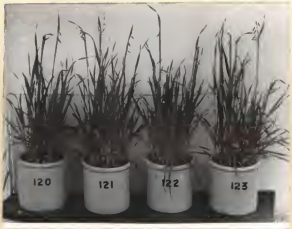
Photograph 15. Oats Growing on Leon Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. April 1, 1954.

Photograph 16. Millet Growing on Leon Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. May 20, 1954.

Photograph 17. Hubam Growing on Leon Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. April 1, 1954.

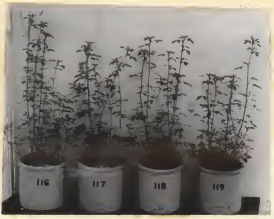
Photograph 18. Soybeans Growing on Leon Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. May 20, 1954.

Photograph 19. Oats Growing on Red Bay Fine Sandy Loam to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. March 1, 1954.



Photograph 20. Oats Growing on Red Bay Fine Sandy Loam to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. April 1, 1954.

Photograph 21. Millet Growing on Red Bay Fine Sandy Loam to Which Sulfur Was Added at the Rates of 0, 1.76, 7.44, and 29.76 lbs/acre, Left to Right. May 20, 1954.



Photograph 22. Hubam Growing on Red Bay Fine Sandy Loam to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. April 1, 1954.

Photograph 23. Soybeans Growing on Red Bay Fine Sandy Loam to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. May 20, 1954.

Photograph 24. Oats Growing on Rutlege Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. March 1, 1954.

Photograph 25. Oats Growing on Rutlege Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. April 1, 1954.

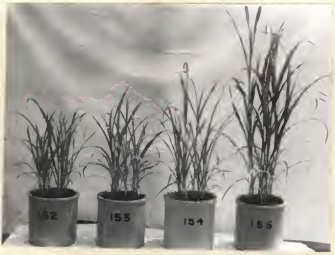
Photograph 26. Millet Growing on Rutlege Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. May 20, 1954.

Photograph 27. Hubam Growing on Rutlege Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. April 1, 1954.

Photograph 28. Soybeans Growing on Rutlege Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. May 20, 1954.

Photograph 29. Oats Growing on Blanton Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. March 1, 1954.

Photograph 30. Oats Growing on Blanton Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. April 1, 1954.



Photograph 31. Millet Growing on Blanton Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. May 20, 1954.

Photograph 32. Hubam Growing on Blanton Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. April 1, 1954.

Photograph 33. Soybeans Growing on Blanton Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. May 20, 1954.

Photograph 34. Oats Growing on Gainesville Loamy Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. March 1, 1954.

Photograph 35. Oats Growing on Gainesville Loamy Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. April 1, 1954.

Photograph 36. Millet Growing on Gainesville Loamy Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. May 20, 1954.

Photograph 37. Hubam Growing on Gainesville Loamy Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. April 1, 1954.

Photograph 38. Soybeans Growing on Gainesville Loamy Fine Sand to Which Sulfur Was Added at the Rates of 0, 1.86, 7.44, and 29.76 lbs/acre, Left to Right. May 20, 1954.

Photograph 39. Oats Growing on Sections of a Virgin Blanton Fine Sand Profile: 0-6, 6-18, 17-30, and 30-42 inches, Left to Right. April 5, 1955.

Photograph 40. Soybeans Growing on Sections of a Virgin Blanton Fine Sand Profile: 0-6, 6-18, 18-30, and 30-42 inches, Left to Right. June 16, 1955.

Photograph 41. Oats Growing on Sections of a Virgin Gainesville Loamy Fine Sand Profile: 0-6, 6-18, 18-30, 30-42, and 42-54 inches, Left to Right. April 5, 1955.

Photograph 42. Soybeans Growing on Sections of a Virgin Gainesville Loamy Fine Sand Profile: 0-6, 6-18, 18-30, 30-42 and 42-54 inches, Left to Right. June 16, 1955.

BIOGRAPHICAL ITEMS

Frank David Bartlett was born November 6, 1928, at Clarksburg, West Virginia. He attended Morgan Grade School and was graduated from Roosevelt-Wilson High School. He received a diploma from Potomac State School of the West Virginia University and a Bachelor of Science and a Master of Science degree in Agriculture from the West Virginia University.

He entered the Graduate School at the University of Florida in 1951 and held the position of Laboratory Assistant in the Florida Agricultural Experiment Station. He expects to obtain the degree of Doctor of Philosophy in August 1955. He was employed by the United States Department of Agriculture in the Soil Conservation Service. He is a member of Sigma Xi and Gamma Sigma Delta honoary societies.

This dissertation was prepared under the direction of the chairman of the candidate's supervisory committee and has been approved by all members of the committee. It was submitted to the Dean of the College of Agriculture and to the Graduate Council and was approved as partial fulfillment of the requirements for the degree of Doctor of Philosophy.

August, 1955

C. V. Noble
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